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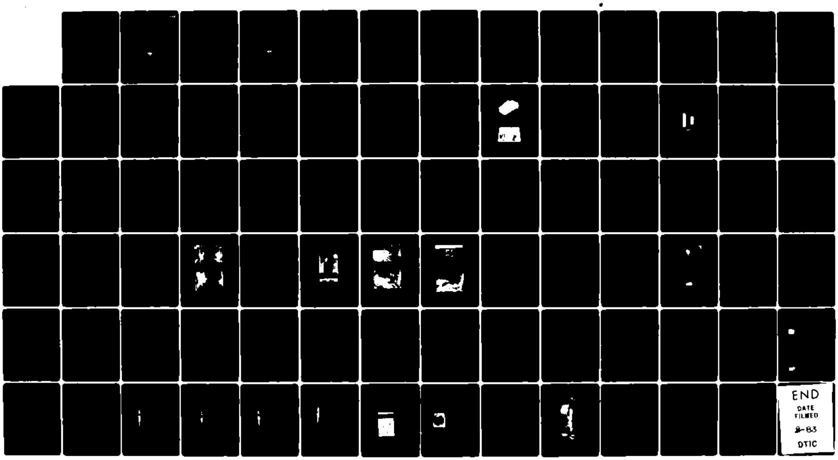

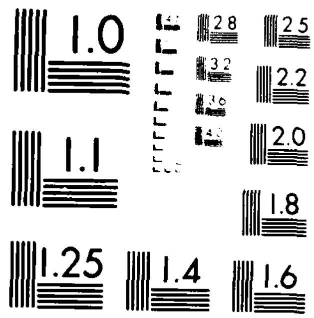

MICROCOPY RESOLUTION TEST CHART

NAVSEA MT REPORT S-587-79

FEBRUARY 1982

POWDERED METAL SINTERING OF JET VANES

AD A 124 032

A PROJECT OF THE
MANUFACTURING TECHNOLOGY PROGRAM
NAVAL SEA SYSTEMS COMMAND

MANUFACTURING TECHNOLOGY

NAVAL WEAPONS CENTER
NWC
CHINA LAKE, CALIFORNIA

FINAL REPORT

Naval Weapons Center
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This report presents the successful efforts to use powdered metallurgy techniques in manufacturing jet vanes from sintered and copper infiltrated tungsten. Isostatic pressing of the powdered tungsten to net shape imparts sufficient hardening to allow handling and final shaping by use of conventional sanding belts. Relatively high pressures increase density to a predictable shrinkage during sintering. Only minor machining after copper infiltration is needed, reducing material usage and labor.

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FOREWORD

This Project Final Report is provided in accordance with NAVMATINST 4800.36D, Manufacturing Technology Program. The project was approved and funded by the Naval Sea Systems Command (NAVSEA) Work Request No. N0002379WR9K242 and assigned Manufacturing Technology Project No. DNS 00587. The lead laboratory was the Naval Weapons Center (NAVWPNCEN), China Lake, CA, Code 3273, with M. J. Ripley-Lotee and S. M. O'Neil sharing technical management. Principal investigators for the Battelle Columbus Laboratories, which provided services under Contract No. N00123-80-C-0038, were J. H. Peterson and K. E. Meiners.

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EXECUTIVE SUMMARY

The Harpoon and Anti-Submarine Rocket (ASROC) Vertical Launch Programs employ a Thrust Vector Control (TVC) module during launch. Vector control is by use of jet vanes to direct the flow of exhausting rocket gases of extremely high temperature, requiring heat-resistant vanes. Specifications are for sintered and copper-infiltrated tungsten.

During the research and early development stages, vanes were produced from a rectangular billet of previously sintered and copper-infiltrated tungsten from which almost 50% of the material had to be removed. Machining of the hardened material required special tools and was time consuming.

Powdered metallurgy (PM) techniques of isostatically pressing other metals into net shapes had successfully demonstrated that material waste and machining time could be reduced, resulting in cost-savings. Tungsten as it is claimed from ores is a natural metal powder. It must undergo further processing to be useful. If it could be formed into net shapes using the PM techniques, it was expected that cost-savings would result.

It was early discovered that machining time was not significantly reduced by using a processed (sintered and copper-infiltrated) net shape. It was determined that labor costs could be reduced best if the material could undergo shaping in the green or unprocessed state after pressing.

Using a relatively high pressure to form the net shape allowed enough strength to be imparted to the material so that is could be handled and shaped into final form by use of a conventional belt sander; the pressed density made the shrinkage during the following sintering stage highly predictable.

Firing tests of the experimental vanes were conducted at the Naval Weapons Center and analyses of the vanes indicated that the new techniques would produce vanes equal to, and in some instances better than, vanes produced under the earlier methods.

A cost analysis for the vanes produced under the MT project indicated that the per-vane cost in quantities of 5,000 would be about $380 as compared to the approximate $550 cost under the earlier established method.

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FINAL REPORT

on

FABRICTION OF COPPER INFILTRATED TUNGSTEN JET VANES

to

COMMANDER, NAVAL SEA SYSTEMS COMMAND

(SEA 0523)

WASHINGTON, D. C. 20362

February 1982

By M. J. Ripley-Lotee and S. M. O'Neil

Introduction

The objective of this program is the establishment of a net-shape powder metallurgy procedure to reduce the production cost of copper-infiltrated tungsten jet vanes. Several rocket motors employ jet vanes as the thrust vector control technique. In order to survive the extremely high temperature of the rocket motor exhaust, the vanes are constructed as a two-component metal composite. A porous tungsten skeleton, which provides high-temperature strength, is prepared by powder metallurgy to a controlled porosity level. This skeleton is subsequently infiltrated with molten copper. In operation, the hot gas of the rocket causes the copper to be melted, vaporized, and expelled from the tungsten skeleton. The cooling effect of the phase changes and the thermal conductivity of the copper serve to keep the temperature of the vane at a tolerable level.

Cost reduction of the vane hinges squarely on the ability to minimize the amount of tungsten used to fabricate the vane, and the amount of machining required overall, but particularly on the sintered part. Except in rare situations where arc cast material is used, tungsten is fabricated by powder metallurgy (P/M) techniques because powder is the primary product form. Most P/M tungsten is fabricated into simple mill shapes which are either used as-sintered or further processed by various metalworking procedures. There has been little emphasis on fabricating net shapes from tungsten powder with the exception of certain liquid phase sintered tungsten parts. The liquid phase sintered product is unacceptable for jet vanes because of the low melting point of the grain boundary phase.

During this program a process was developed which utilizes simple, rapid machining of a pressed-to-near-net shape prior to the sintering and infiltration operations. Very little conventional machining of the jet vane is required to meet the final dimensions.

Included in this report are the conclusions of a producibility study, the results of the fabrication studies conducted, a manufacturing process description, an inspection and test plan, and a cost estimate analysis for producing the vanes.

Producibility Study

Prior to the experimental work on this program, a producibility study was conducted to determine the most cost effective method of producing the copper-infiltrated tungsten jet vane. The two areas which most influence the cost of the vane are machining and the tungsten material. The sintering and infiltration costs will be approximately the same whether the shape of the blank for the vane is a rectangular block or a near-net shape.

Although tungsten is a relatively expensive material, the cost advantage of producing a near-net shape compared to a rectangular block is not as great as might be expected. Assuming a cost of $40/kg for tungsten, the cost of the material in a rectangular blank 0.75 x 3.25 x 4.25 inches with a density of 80 percent of theoretical is $104 (2.6 kg x $40/kg). Producing a near-net shape of the tungsten vane would reduce the weight to about 1.6 kg and result in a savings of about $40 per vane. Since the total machining cost, which is estimated to be about $400, is considerably more than the materials cost, any potential cost reduction would best be achieved by reducing the machining time. The machine shop staff at BCL indicated that it may be more cost effective from the standpoint of conventional machining practice to start the machining operation using a rectangular block instead of a near net shape due to the ease of initial set-up. If this is true, the cost advantage in beginning a conventional machining operation with a near-net shape would be lost.

To minimize conventional machining costs, BCL recommended that most of the machining of the tungsten preform be done in the green condition. The green condition is defined as the state of the part following the pressing operation, but prior to the sintering process. Although this approach had not been tried with tungsten, it has been done with other materials in an effort to reduce or eliminate final machining costs. If the green parts are at a density level reasonably close to the sintered density, the shrinkage during sintering is predictable and close-to-final dimensions can be obtained. In the conventional pressing of P/M tungsten parts before sintering, pressures in the range of 30,000 - 60,000 psi are usually used. These pressures produce parts in the range of 60 to 70 percent of theoretical density depending upon the particle size of the powder. However, higher pressure presses having the pressure capability of up to 100,000 psi required for relatively small parts such as the jet vane are not uncommon. It was believed to be feasible to press the tungsten powder to a near-net shape at high pressure and green-machine all but a few of the critical areas prior to sintering and infiltration. By use of a belt sanding technique using special fixtures to hold the green-pressed shape, nearly all of the surfaces could be rapidly machined. Prior to the sintering operation, the tungsten powder particles are held together only by mechanical interlocking and can be readily broken away by a sanding operation. After sintering, the particles are metallurgically bonded together and considerable energy is required to machine the material. Because tungsten is a relatively hard material, carbide cutting tools are required for machining after sintering. After infiltration, machining is much easier, but good practice still requires carbide tooling.

The fabrication approach recommended above would be most favorable if the green part has sufficient strength for the sanding (machining) operations and the shrinkage during sintering is sufficiently predictable to obtain the required final dimensions on most of the vane surfaces.

Fabrication Studies

This program was divided into three distinct phases.

Phase I - Experimental Fabrication
Phase II - Preprototype Fabrication
Phase III - Prototype Fabrication.

As the work in Phase I evolved, it became apparent that the vane machining concept proposed by BCL should be developed during the first phase rather than deferring this development to the subsequent phases. Therefore, nearly all of the vane fabrication development occurred during Phase I, with only minor changes and modifications to the process during Phases II and III. The fabrication studies discussed in this section include the combined work of all phases of the program.

The vane fabrication studies conducted during this program were divided into five areas.

1. Powder Selection and Processing
2. Vane Fabrication and Machining
3. Infiltration
4. Fabrication of Vanes for Evaluation
5. Inspection.

There was considerable overlap among these areas as work was being conducted on the first three at the same time during most of Phase I. However, each is discussed individually in the following sections.

Powder Selection and Processing

Three different particle size powders were selected for evaluation. These were 0.5-1.5, 3.5-4.0, and 5.0-6.0 micron powders. In addition to these three as-received powders, two blends of these powders were also evaluated in an effort to achieve a higher vibratory density prior to cold isostatic pressing. These five powders and blends of powder were as follows:

1. 0.5 - 1.5 micron
2. 3.5 - 4.0 micron
3. 5.0 - 6.0 micron
4. Blend A:
   50% 3.5 - 4.0 micron
   50% 5.0 - 6.0 micron
5. Blend B:
   12% 0.5 - 1.5 micron
   44% 3.5 - 4.0 micron
   44% 5.0 - 6.0 micron.

The experimental work conducted on these powders consisted of determining the vibratory density of each powder and the densities attained after pressing at various pressures from 60,000 to 150,000 psi. The vibratory densities were determined by weighing the powder in a graduated cylinder after vibrating on a vibratory table.

For the evaluation of green densities, a shape about 0.6 inch in diameter by 4 inches long was used. These bars were isostatically pressed from the powder using a thin natural latex rubber bag. The results, presented in Table 1, showed that two of the powders could be dropped from consideration. The 0.5 - 1.5 micron powder had too low a vibratory density and too low a green density to be considered for use as a vane material. The shrinkage that would have to occur during sintering to achieve the desired final density would be so great that excessive distortion would be the probable result. Blend B did not produce a significant increase in density. Therefore, because of the higher costs of blending with no associated benefit, this powder was also dropped.

The target density for the tungsten skeleton prior to infiltration with copper is 75-83 percent of theoretical; a pressure of 85,000 psi was selected as an appropriate pressure. There would be some shrinkage of the green parts during sintering, which would place those parts pressed at 150,000 psi above the density range prescribed for the vanes. Also, the availability of equipment for pressing above 100,000 psi is limited and the transfer of this technology to a manufacturer at the conclusion of this program would be more difficult. Thus, it did not appear necessary to press at pressures in excess of 100,000 psi.

Those specimens which were pressed at an estimated pressure of 85,000 psi from the three powders selected for further study (Powders 2, 3, and 4) were sintered at 1900°C in vacuum for 1 hour. Two sintering runs were made; the first contained one pressed specimen of each powder and the second contained two specimens of each powder lot. The results are given in Table 2. The densities obtained after sintering were in the middle of the desired range and were considered to be satisfactory for the jet vanes.

TABLE 1. COLD ISOSTATIC PRESSING STUDIES ON TUNGSTEN POWDER

Particle Size, microns	Vibratory Density, percent	Green Pressed Density, percent		
		60,000 psi	85,000 psi[a]	150,000 psi
0.5 - 1.5	18.8		63.8	
3.5 - 4.0	29.2	68.6	75.7	81.9
5.0 - 6.0	30.5	69.6	77.2	83.4
Blend A	28.8		76.6	82.4
Blend B	28.7		75.6	82.2

(a) The indicated pressure of 85,000 psi is an estimate of the pressure used for the cold isostatic studies. The gage actually used for this work was found to give erroneous readings and had failed in service so that the magnitude of the discrepancy in pressure could not be determined. A comparison of the density obtained from the same powders using a calibrated pressure gage indicated that this initial work had been performed at approximately 85,000 psi.

TABLE 2. DENSITY OF TUNGSTEN AFTER SINTERING AT 1900°C in VACUUM FOR 1 HOUR[a]

Particle Size, microns	Density, percent
	Average of three specimens
3.5 - 4.0	80.4
5.0 - 6.0	80.4
Blend A	79.7

(Specimens had been cold isostatically pressed at 85,000 psi prior to sintering.

In the final selection of the powder to be used in fabricating the jet vane, the following factors were considered: infiltration characteristics, tensile properties, and machinability in the green condition. This selection was not made until the fabrication of the four vanes for Phase I evaluation was initiated.

Vane Fabrication and Machining Studies

The first step in the fabrication of the vane shape was to prepare tooling to make a shaped pressing bag. This bag had the shape of the vane but was sufficiently oversized so that after pressing the resulting shape would have just enough stock for clean-up during the green machining operation. For the first trial, a polyvinyl chloride pressing bag with a wall thickness of about 0.10 inch was used. During loading of the powder, the bag was supported on the exterior to retain the desired shape. After loading with powder, a closure was inserted and the powder was de-aired. By de-airing the powder, there is sufficient external pressure to maintain the shape of the part during the handling operations prior to pressing. After pressing, it was noted that fracturing of the part had occurred. The bag had adhered tightly to the surface of the part and springback of the bag to its normal position after pressing had caused breaking of the part. In the second trial, a bag with less strength was used, a natural latex bag with a thickness of about 0.035 inch. This pressing bag performed very satisfactorily and the desired vane configuration was obtained. This type of bag was then used for the fabrication of parts in Phases I and II.

During Phase I, the loading fixture used to hold the bag during loading was in one piece. The bag had to be withdrawn from the loading fixture through the top opening; pulling on the bag to accomplish this often caused distortion of the de-aired powder within the bag. Evacuation of the air within the bag is normally sufficient to minimize distortion during normal handling, but often the bag was difficult to remove. During Phase II, an improved vacuum loading fixture was fabricated to to support the latex bag used for pressing the powder. This fixture, shown in Figure 1, permitted distortion-free removal of the bag after loading and evacuation. The urethane supports for the bag were divided along the natural parting line of the vane and after removal of the top, bottom, and outer housing of the fixture the urethane supports were separated, leaving the loaded bag ready for pressing. Although this was a marked improvement in the loading procedure, alternative methods of accomplishing this step were investigated that would be more production oriented. During Phase III, a polyvinyl chloride bag which was thinner (0.05 inch thick) than the first bag of this type used in Phase I was evaluated. The bag, which was also made on the shaped tooling, was supported in a perforated sheet metal cage to maintain the desired vane shape. After loading, the bag was clamped at the top with metal bars to obtain a seal and the powder was not de-aired. The desired shape of the powder in the bag was maintained during pressing by leaving the bag in the perforated metal cage. The bag was sufficiently thin so that fracturing of the green part did not occur upon release of the pressure after pressing. These changes significantly reduced the time to prepare the powder for pressing and also reduced the complexity of the operation. The majority of the vanes produced during Phase III were pressed using this procedure.

Machining of the green vane was then evaluated. As stated previously, the approach evaluated was to position the green part in specially designed fixtures and remove the excess stock by belt sanding. The first parts of the vane to be machined are the narrow top and bottom surfaces. However, prior to positioning the vane in the fixture for these operations, the high spots on one side of the part (two large flat faces) are smoothed by sanding in a fixture to provide initial reference surfaces. This is not the final stock removal on these faces. The part is then clamped in the fixture and the excess material removed from the top and bottom surfaces by belt sanding. Machined dimensions of the part are controlled by tungsten carbide inserts, brazed to the surface of the fixture, acting as stops to limit the movement of the part into the belt. A photograph of this fixture is shown in Figure 2.

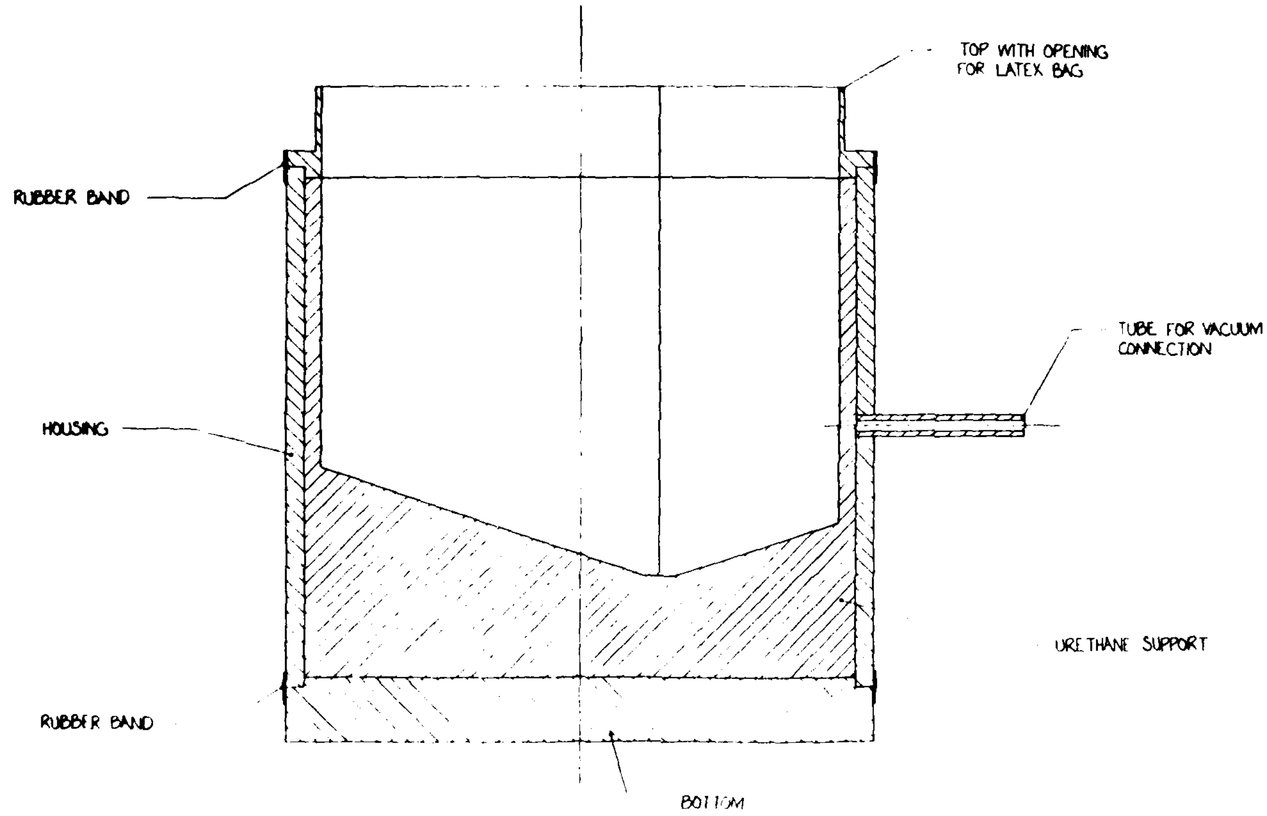

FIGURE 1. POWDER LOADING FIXTURE

After machining of the top and bottom surfaces, the vane is placed in a fixture for machining of the flat faces on the side of the part. This fixture is shown in Figure 3. The previously prepared flat faces and the top and bottom surfaces are used as reference surfaces.

After one side (two flat faces) has been machined, the part is repositioned in the fixture with the other side up. The cradle that supports the vane on the flat faces is raised slightly to compensate for the material removed from the first side machined (to permit stock removal on the unmachined faces). The operation is then repeated.

In Phase I, attempts were made to control both the overall height of the vane and the length of the vane by green machining. Due to minor distortion during sintering, it was found to be more desirable to leave some excess material on the bottom surface and the ends of the vane for machining to final dimensions after sintering and infiltration. The dimensions of the vane after green machining are slightly oversized to provide for the small amount of shrinkage that occurs during sintering.

Sintering of the vane was done in vacuum at 1900°C for 1 hour. The first vane to be sintered was made with 5.0 - 6.0 micron powder. Although the expected density was obtained (about 80 percent of theoretical), a small crack was noted after sintering. Additional vanes prepared from both the 3.5 - 4.0 and 5.0 - 6.0 micron tungsten powders were sintered with no cracking observed. The shrinkage of the parts during sintering was about 0.02 inch/inch and was reproducible.

Infiltration

Infiltration studies were conducted with three types of powder that had been cold pressed at 85,000 psi and sintered in vacuum at 1900°C for 1 hour. The powder used had particle sizes of 3.5 - 4.0 microns, 5.0 - 6.0 microns, and a blend of equal amount of these two powders (Blend A). The specimens were bars about 0.6 inch in diameter by 4.0 inches long. The first infiltration experiment was conducted

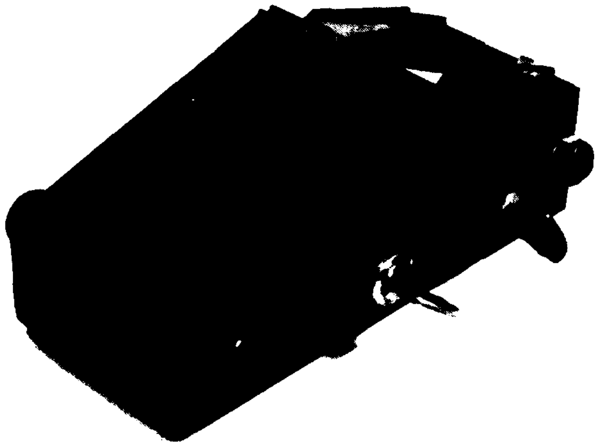

FIGURE 2. FIXTURE FOR MACHINING OF TOP AND BOTTOM SURFACES

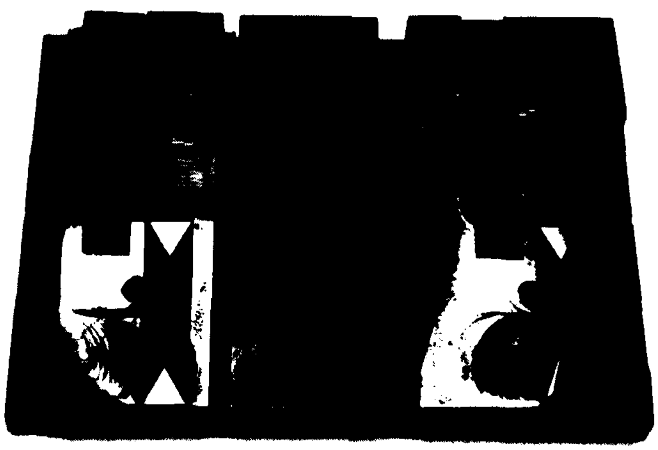

FIGURE 3. FIXTURE FOR MACHINING THE SIDES OF THE VANE

by placing the pressed and sintered bars horizontally on copper sheet in a graphite boat. The infiltration was accomplished by heating to 1200°C in hydrogen, holding for 1 hour, and furnace cooling. The percent infiltration on these bars ranged from about 80 to 90 percent. The desired infiltration is a minimum of 90 percent. Examination of the bars indicated that the exterior of the bars was well infiltrated, but the center portion was very poorly infiltrated. The same experiment was repeated with the bars placed in a vertical position and the copper surrounding only the bottom end of the bar. The results were very similar. It was apparent that the copper coated the exterior surface of the bar almost immediately upon melting of the copper and gases trapped in the interior of the bar resisted further infiltration. To prevent this rapid coating of the bar surface by copper, a brazing stop-off was applied to the exterior surface, leaving only the bottom of the bar open to the molten copper. Infiltration was attempted again with the bars in the vertical position, with much improved results. The percent infiltration on duplicate bars of the three powder types was well in excess of 90 percent, with 92.6 percent the lowest value obtained.

The bars from the last run described above were machined into tensile bars. These bars had a reduced gage section of 0.25 inch in diameter by 1.25 inches long. The tensile tests were performed on an Instron testing machine at a strain rate of 0.005 in./in. per minute. The tensile strengths obtained were very consistent in the range of 61,500-68,500 psi, but below the required minimum of 80,000 psi. Examination of these bars after fracture showed that a portion of the center was still not filled with copper. This would be expected to materially affect the tensile strength of the bar since, although it amounts to only about 3 percent of the total volume of the bar, it is a significant portion of the reduced gage section in the tensile specimen.

In seeking to improve the infiltration behavior, inquiries were made outside BCL to determine the techniques used in industry for infiltration of tungsten. The primary suggestion received was to rapidly heat, minimize the time at temperature, rapidly cool. It was also indicated that the infiltration process was difficult to control and required some amount of "art" to achieve full infiltration. Three different experiments based on these suggestions were then performed. The first of these experiments was the rapid heating to 1230°C in hydrogen, followed by a short dwell time at temperature and then rapid cooling. Another infiltration run was made in vacuum at 1175°C. The third run was begun in 1 atmosphere hydrogen (up to a temperature of 1050°C), then the furnace was evacuated to about 100 microns pressure of hydrogen gas and the temperature increased to 1230°C for the copper infiltration. None of these variations altered the general pattern of the location of the copper in the porous tungsten, leaving the central portion uninfiltrated.

At this time, work was progressing satisfactorily with the pressing, machining, and sintering of the vane shape. Therefore, it was decided to use the actual vane configuration in the infiltration studies rather than the 0.6-inch diameter bars used in the experiments described above. A vane made with 5.0 - 6.0 micron powder that had been sintered at 1900°C was infiltrated with copper in a hydrogen environment at 1230°C for 0.5 hour. After infiltration, the vane was sectioned and areas were found that were not infiltrated.

Two additional vanes were then prepared, one from 3.5-4.0 and one from 5.0-6.0 micron tungsten powders. After machining and sintering, these vanes were infiltrated using a modified infiltration cycle. This cycle consisted of heating to just below the melting point of the copper (1040°C) in hydrogen and holding for 1.5 hours to assure clean-up of the tungsten skeleton, followed by increasing the temperature to 1260°C for 0.5 hour for the infiltration. Density measurements and examination of the vanes by sectioning indicated that the vanes had been uniformly infiltrated. Figure 4 shows a cross section of the vane described previously which was not fully infiltrated and a representative cross section of one of the vanes processed with the modified infiltration cycle. Metallographic examination also indicated that excellent infiltration of the porosity had been obtained. The microstructures of the sintered and infiltrated material made from 3.5 - 4.0 micron powder and the 5.0 - 6.0

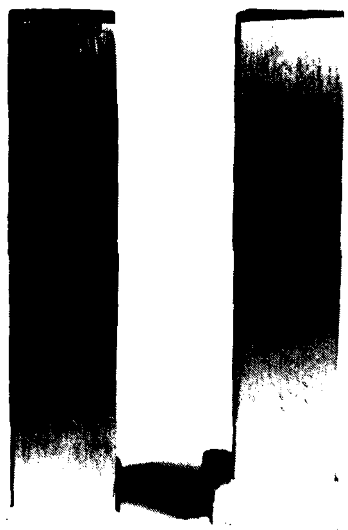

FIGURE 4. CROSS SECTIONAL VIEW OF INFILTRATED VANES

The section on the left is well infiltrated. Two vertical bands in other vane are not infiltrated.

micron powder are shown in Figure 5 and 6, respectively. Nearly all the pores have been filled and there is some shrinkage, about 5 percent, of the copper during solidification which would account for some of the unfilled areas. The tensile strength of these materials was also much improved over that of the previously evaluated poorly infiltrated material. Two tensile bars were machined from each of the vanes and tested at a strain rate of 0.005 in./in./min. The diameter of the reduced section of the tensile specimens was 0.187 in. The results of these tests and the percent of infiltration are given in Table 3. Examination of the fracture sections of the bars also showed full infiltration.

The results obtained from sintered and infiltrated vanes fabricated from both of the powder particles sizes meet the requirements specified for the vanes. The percent infiltration is greater than 90 percent and the tensile strength is in excess of 80,000 psi. From these results, the selection of the 3.5-4.0 micron tungsten powder as the most suitable material for a production process was made for the following reasons:

1. The tensile properties for this powder were slightly higher.
2. Cracking had been observed in one of the 5.0-6.0 micron powder vanes.
3. The 3.5-4.0 micron powder is a more commercially available powder than the 5.0-6.0 micron powder.

To further characterize the material selected for fabrication of the vanes, a chemical analysis was made of the infiltrated tungsten made with the 3.5-4.0 micron powder. The analysis, given below, showed low impurity levels.

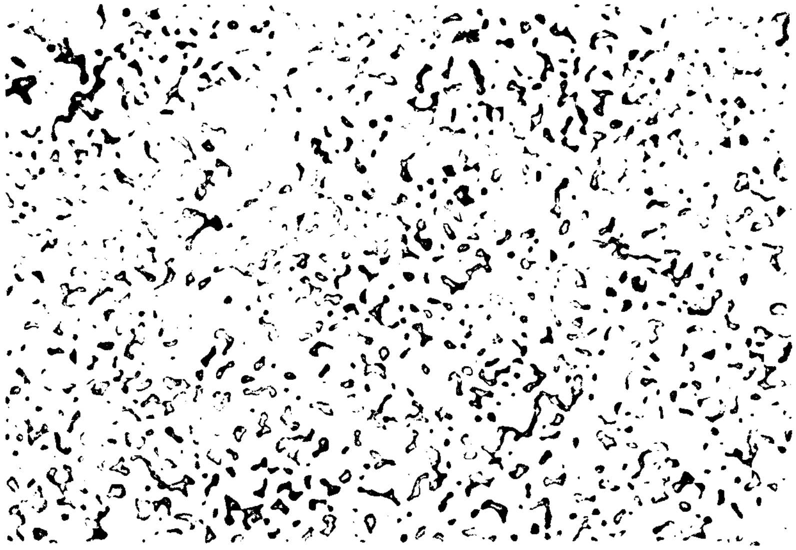

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FIGURE 5. 3.5-4.0 MICRON TUNGSTEN AFTER SINTERING AND INFILTRATION IN HYDROGEN AT 1040°C FOR 1.5 HOUR, THEN 1260°C FOR 0.5 HOUR

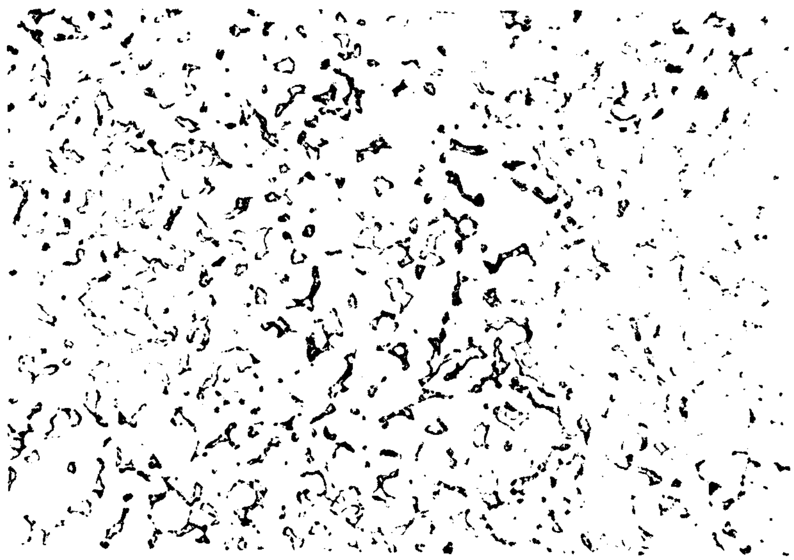

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FIGURE 6. 5.0-6.0 MICRON TUNGSTEN AFTER SINTERING AND INFILTRATION IN HYDROGEN AT 1040°C FOR 1.5 HOURS, THEN 1260°C FOR 0.5 HOUR.

TABLE 3. TENSILE RESULTS OF COPPER-INFILTRATED TUNGSTEN

Powder Size, microns	Sintered g/cc	Density, percent	Percent Infiltration	Ultimate Tensile Strength,[a] psi
3.5 - 4.0	15.54	80.5	96.0	111,600
				109,800
5.0 - 6.0	15.34	79.5	92.5	108.700
				99,600

(a) Two test specimens for each powder type.

Element	Impurity Level, ppm
Si	100
Fe	100
Mg	100
Pb	100
Al	100
Ca	100
Ag	<100
Ni	<100

Fabrication of Vanes for Evaluation

As a result of the experimental work on this program, a fabrication procedure for producing the copper-infiltrated tungsten jet vane was established. A brief description of the fabrication procedure is given below:

1. Press the tungsten powder (3.5-4.0 micron particle size) to near-net shape at 80,000 psi.
2. Machine the pressed part by belt grinding.
3. Sinter in vacuum for 1 hour at 1900°C.
4. Infiltrate with copper at 1260°C for 0.5 hour after a hold at 1040°C for 1.5 hours.
5. Inspect for density, percent infiltration, and internal quality.
6. Machine base to obtain final height, drill and tap holes in the base, and radius the ends of the vane.
7. Inspect for surface quality using dye penetrant inspection.
8. Record part dimensions.

A detailed description of the entire fabrication process is covered in Appendix A, Manufacturing Process Description.

During the three phases of the program, vanes were produced for evaluation by BCL and by NWC. The quantities produced were as follows:

Phase I - 2 vanes for evaluation at NWC
2 vanes for evaluation at BCL
Phase II - 6 vanes for evaluation at NWC
Phase III - 32 vanes for evaluation at NWC.

Throughout the program, certain modifications were made to some of the fabrication procedures to increase the efficiency of the operation or to improve the quality of the finished vane. One modification was made in the procedure for obtaining the density of the sintered vane prior to infiltration. Initially, the sintered vane had been coated with paraffin wax to seal off the pores during immersion in water. Coating with the paraffin wax is time consuming and often a small hole is left in the coating, which results in the filling of some of the pores with water. This results in erroneous density determinations. Removal of the wax and cleaning the surface of the vane prior to spraying with the stop-off used during infiltration was also undesirable. Therefore, because the volume of the sintered and infiltrated vane is the same, the volume determined after infiltration was used to determine the density of the vane after sintering. The excess copper around the base of the vane after infiltration was removed by sawing and/or filing and the remainder by leaching in dilute nitric acid. Because the pore size of the tungsten skeleton is so small, only a superficial removal of the copper from the tungsten results from the leaching operation.

During Phase I, the infiltration of the sintered vane had been done in a furnace that allowed infiltration of only one vane at a time Although this would have been satisfactory for Phase II, it would not have been for the larger number of vanes required in Phase III. Therefore, another furnace was modified for this operation to enable three vanes to be infiltrated at one time. Although the fabrication rate was increased, it was still a batch operation and did not simulate a production condition such as a continuous operation through the furnace. No change was made to the infiltration conditions developed in Phase I and the vanes produced during Phase II exceeded the minimum infiltration requirement of 90 percent of the porosity filled with copper. When larger quantities were produced during Phase III, the percent infiltration showed more variation and was often below 90 percent. These lower values were generally only 2 to 3 percent below the 90 percent level. Processing these vanes through the infiltration process a second time did not improve the percent infiltration. The results of the tensile tests on the infiltrated tungsten were also more erratic than in the experimental work during Phase I. For example, tensile specimens taken from the same vane gave values of 74,750 psi and 104,800 psi. The one test falls below the required minimum of 80,000 psi. Examination of the fracture surface of the low test showed a small area that was not infiltrated, which would initiate fracture at a lower stress level. Although this vane showed a percent infiltration well above the required 90 percent (94.2 percent), this would still allow for small isolated areas that were not filled with copper.

Results from the Phase I work indicated that dimensional control in two locations could be improved during Phases II and III. The first of these was the relation of the two narrow surfaces on the top of the vane to the base. Because the base must be flat and some distortion of this surface did occur during sintering, it was machined by grinding after infiltration. Initially, the vane was clamped between two blocks which had been machined to match the two angles on the sides of the vane. Because these angles are quite shallow, proper alignment was difficult to maintain. Therefore, it was decided to use the long narrow surface on the top of the vane as the reference surface. A fixture was made to support the vane using this surface as a reference prior to clamping it between the two side support blocks. This modification has worked very well and makes the set-up of the vane for subsequent operations in other machines rapid and consistent.

The other improvement in dimensional control concerned the length of the vane. In Phase I, the length of the vane showed some variability because the machining to length was done in the green condition prior to sintering. Due to minor differences in the shrinkage during sintering, the length of the vane was left slightly oversized and machined to final length after sintering, infiltration, and machining of the base, including the holes in the base. A fixture was designed and fabricated that would locate the leading edge of the vane relative to the 0.501-inch diameter hole in the base. The trailing edge is then located 4.000 inches from the leading edge. This machining fixture uses the same approach as the others: belt sanding to obtain the desired stock removal with carbide stops to limit the movement of the fixture into the belt. Originally, the fixture was designed so that the radius on both the leading and trailing edges could be machined using the fixture. The fixture used 0.125-inch diameter carbide rods so that the ends could be dressed to this radius on a belt sander. However, these rods were so small that appreciable wear occurred after limited use. Therefore, the fixture was modified using four carbide inserts on each end of the fixture so that the vane could be machined to length with negligible wear of inserts. After removal from the fixture, the leading and trailing edges of the vane were dressed on a belt sander to obtain the desired radius.

In all three phases, the 0.200 inch flat on the top surface and final machining of the bottom to give the desired height was done by conventional grinding. The 0.501-inch-diameter hole in the base was machined by drilling and reaming with carbide tooling. The two smaller holes in the base were drilled and tapped using conventional high speed steel tooling. No unusual problems were encountered in the machining of the vanes even though the material is relatively difficult to machine and the wall thickness of the 0.501-inch-diameter hole is quite thin along the sides of the vane.

Inspection

During fabrication of the vanes, an inspection procedure was followed to assure that the vanes met the quality standards required. After infiltration, the sintered density, the infiltrated density, and the percent infiltration were determined. The vanes were then radiographed to inspect for internal defects. A Cesium 137 source was used, and two different speeds of film were placed beneath the vanes so that both the thin sections and the thick section on each vane would have the proper exposure for inspection. The vanes were then final machined. Following machining, dye penetrant inspection was made for surface defects and cracks. Because brazing stop-off is used on the exterior of the vane during infiltration, a thin skin on the surface of the vane is not infiltrated with copper. This makes inspection more difficult, but defects such as cracks, voids, laps, or tears should be evident. The results of these inspections were recorded on the data sheet which accompanied each vane during fabrication and inspection. A dimensional inspection of the vane was also made and the measurements recorded on a drawing of the part, which is attached to the data sheet. Copies of the data sheet and the dimensional inspection drawing are included with the Inspection and Test Plan, Appendix B.

Cost Estimate Analysis

In the past NWC obtained jet vanes by purchasing billets of sintered and copper infiltrated tungsten and fabricating the vanes from the billets. The billets were purchased from Teledyne Wah Chang Corp., a known source of high quality tungsten products. Because the vanes are used in the rocket motor exhaust, it was necessary to acquire the best quality of material available in order to prevent jeopardizing the vertical launch program. Under that previous process, materials and sintering for 30 vanes each cost $364, machining was $131, and quality control cost $51, for a total of $546 per vane. Cost figures are from the last vanes fabricated before the MT project. It may be compared to the following cost analysis of fabricating with the new technology.

A cost estimate analysis was conducted for the production of 1,000, 5,000, and 10,000 jet vanes. It was assumed in this analysis that a manufacturer currently making copper-infiltrated tungsten components would be the producer. Therefore, no new equipment would be required, and the production of the jet vanes would fit into the normal production schedule. Costs have been estimated for the labor and supervision operations, equipment/facility use and maintenance, materials, and specialized tooling. No significant differences were found in the labor operations or materials for the number of parts produced, but improvements in yield and lower tooling costs were evident with increasing numbers produced. No allowance for scrap generated during the production of the vanes was made in this analysis.

The cost analysis is outlined in Tables 4, 5, 6, and 7. The summary of costs for the production of jet vanes, Table 7, indicates that if a 15 percent profit is assumed, the costs per vane at production levels of 1,000 vanes is $401.16, at 5,000 vanes is $381.22, and at 10,000 vanes is $372.08.

Immediate cost reduction of the vane can be most likely achieved by relaxing the dimensional tolerances and inspection requirements on the vane so the expense of the machining and inspection operations can be reduced. A long range approach for reducing the cost of the vanes would be to construct a specialized production facility to produce large quantities of vanes. This would result in lower labor costs, particularly in the sintering and machining operations.

Contractor's End-of-Project Demonstration

An end-of-project demonstration was conducted at the contractor's facility 15 September 1981. The demonstration was well received and elicited expressions of interest in the process by members of industry. BCL's Conference Report on Manufacturing Technology Demonstration, Fabrication of Copper Infiltrated Tungsten Jet Vanes, is included as Appendix C to provide more detail on the demonstration.

TABLE 4. MATERIALS COST

	Cost per vane
Tungsten powder	
1.5 kg at $40/kg	$60
Copper for Infiltration	1
Sanding Belt	4
Miscellaneous Materials	2
Plastisol for Pressing Bag	
Stop-Off Spray	
Nitric Acid	
Total Cost per Vane	$67

TABLE 5. TOOLING COSTS

	Number of Vanes Produced					
	1000		5000		10000	
	Tools Required	Cost	Tools Required	Cost	Tools Required	Cost
Tooling for Pressing of Vanes						
Forms for Dipping of Bag	2	$ 600	3	$ 900	4	$ 1,200
Perforated Metal Loading Cage	8	800	12	1,200	16	1,600
Tooling for Green Machining						
Fixture for *Top and Bottom Surfaces*	2	1,200	4	2,400	6	3,600
Fixture for First 2 sides	2	2,000	4	4,000	6	6,000
Fixture for Second 2 sides	2	2,000	4	4,000	6	6,000
Tooling for Final Machining						
Fixture for Conventional Machining	2	1,000	4	2,000	6	3,000
Fixture for Sanding to Length	2	1,600	4	3,200	6	4,800
Total Cost		$9,200		$17,700		$26,200
Cost per Vane		$9.20		$3.54		$2.62

TABLE 6. OPERATING COSTS FOR PRODUCTION OF JET VANES

Labor Operation	Time, hr per Vane	Equipment/Facility Use and Maintenance, Cost per hr	Equipment/Facility Use and Maintenance, Cost per vane
Pressing Bag Fabrication	0.30	$ 5	$ 1.50
Loading of Part	0.30	5	1.50
Pressing of Part	0.25	20	5.00
Green Machining	0.30	5	1.50
Sintering	1.00	20	20.00
Infiltration	0.30	20	6.00
Removal of Excess Copper	0.30	5	1.50
Density Determination	0.50	5	2.50
Radiography	0.30	5	1.50
Machining of Base and Flat	0.30	5	1.50
Machining of Holes	1.00	5	5.00
Dye Penetrant Inspection	0.25	5	1.25
Dimensional Inspection	0.30	5	1.50
Supervision	0.85	5	--
Totals	6.25 hr		$50.25
<u>Labor Cost</u>			
Direct Labor and Supervision (DLS) (6.25 hr x $15.00)			93.75
Indirect Labor (25 percent of DLS)			23.44
Fringe Benefits (25 percent of DLS)			23.44
General Overhead (50 percent of DLS)			46.87
Total Labor Cost			$187.50
<u>Equipment/Facility Use and Maintenance</u>			
Total Cost from Above			$ 50.25

TABLE 7. SUMMARY OF COSTS FOR PRODUCTION OF JET VANES

Cost Item	Cost per Jet Vane, dollars 1,000 Vanes	 5,000 Vanes	 10,000 Vanes
Total labor cost	187.50	187.50	187.50
Equipment/facility use and maintenance	50.25	50.25	50.25
Materials cost	67.00	67.00	67.00
Tooling cost	9.20	3.54	2.62
Total production cost	313.95	308.29	307.37
Cost per vane at yield indicated	348.83 (90%)	331.49 (93%)	323.55 (95%)
Total cost including 15 percent profit	401.16	381.22	372.08

Motor Static Firing Tests

Three tests were conducted on the vanes produced with the manufacturing technology process. The tests are necessary to determine whether the vanes can perform in the environment of the rocket motor exhaust for the required length of time. In each case the vanes were equal to or somewhat better than vanes produced by the earlier method. The three firing test reports are included as Appendix D and fully support the acceptability of the use of the new technique in the manufacture of the jet vanes.

Conclusions

During this program a unique P/M processing approach has been developed to produce the copper-infiltrated tungsten jet vane. This process minimizes the input material by pressing the vane to near net shape. By pressing at a relatively high pressure, adequate strength is imparted to the vane to allow machining in the green condition, and a high enough density is obtained so that the shrinkage that occurs during sintering is very predictable. By machining in the green condition using an abrasive belt, the machining time for most of the surfaces on the vane is reduced to 10-15 minutes. Only limited conventional machining is required after sintering and infiltration.

Inspection of the vanes indicated that high quality vanes meeting the contract requirements could be produced by this process. Firing tests performed at NWC indicated that the vanes produced by this process performed equal to or better than vanes made by the conventional process evaluated previously. This improvement in performance is probably due to the finer particle size of powder used in this fabrication process rather than a result of the process itself.

A cost estimate analysis for the production of the vane by this process gave an estimated cost of approximately $380 per vane in quantities of 5,000 parts.

Recommendations and Implementation

It is recommended that the procedures and processes developed during this manufacturing technology project be adopted for use during the following phases of the vertical launch program and that the technology be disseminated, to the extent feasible and prudent, to other weapons systems managers that could profit from the techniques established.

An Implementation Plan dated 9 September 1981 has been forwarded, and no changes in the plan are foreseen. Implementation procedures will be activated for the engineering development phase of the vertical launch program for ASROC and HARPOON missile systems.

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APPENDIX A

MANUFACTURING PROCESS DESCRIPTION FOR COPPER INFILTRATED TUNGSTEN JET VANES

The experimental work in Phases I, II, and III of this contract has resulted in a process for the fabrication of copper infiltrated tungsten jet vanes. This process is sufficiently well defined that a detailed description of the operations to produce the vane has been prepared. The fabrication of the vane is divided into eight distinct process steps. These are:

1. Press the tungsten powder (3.5 - 4.0 micron particle size) to near-net shape at 80,000 psi
2. Machine the pressed part
3. Sinter in vacuum at 1900°C for 1 hour
4. Infiltrate with copper at 1260 - 1280°C for 0.5 hour after a hold at 1040°C for 1.5 hours
5. Inspect for density, percent infiltration, and internal quality
6. Machine base to obtain final height, drill and tap holes in the base, and radius the ends of the vane
7. Inspect again for surface quality using dye penetrant inspection
8. Record part dimensions.

A description of each of these steps is given below. Drawings of the various fixtures and tooling used in producing the vane have been provided as well as photographs showing the various operations.

Cold Isostatic Pressing

For the cold isostatic pressing (CIP) of the tungsten powder into the vane configuration, a thin polyvinyl chloride (PVC) bag and a loading fixture to support the exterior of the bag are required. The PVC bag is made using the dipping form shown on Drawing No. NWC-001-000.

This form has allowance for the change in the volume of the powder from the vibratory packed condition to the sintered condition, machining stock on the pressed part after CIP, and normal shrinkage of the bag during curing of the bag. The bags used on the program were made using a standard plastisol: Mistaflex 413-V-900 clear plastisol obtained from M-R Plastics and Coatings, Maryland Heights, Missouri. The thickness of the bag is approximately 0.05 inch.

The loading fixture which supports the exterior of the bag during loading of the powder is fabricated from 16 gage perforated sheet steel. The loading fixture is made to conform to the exterior of the bag while the bag is supported internally by the dipping form. An assembly of the fixture and the bag is shown in Drawing No. NWC-002-000. After placing the bag into the loading fixture, the assembly is placed on a vibratory table and powder loaded into the bag as shown in Figure 1. After the powder has been loaded and leveled, a foam rubber filler about 1/2 inch thick is placed over the powder to maintain the desired shape of the powder at the top of the bag when the closure is made. Sealing of the top of the bag is accomplished by folding over about 1-1/2 inches of the bag and clamping two metal strips, 1/4 x 1 x 7 inches in size, over this fold with two "C" clamps. A photograph of this assembly is shown in Figure 2. The bag containing the powder is left in the loading fixture to maintain the desired shape during loading into the CIP vessel and during the initial stages of pressing.

The part is then cold isostatically pressed. Because BCL does not have a hydropress large enough to handle the pressing in one operation, the part is pressed to 50,000 psi in one press (8-inch ID) and subsequently pressed to 80,000 psi in a 4-inch ID press. The bag can be reused a number of times before discarding.

Machining of the Pressed Vane Shape

Most of the machining operations are performed with the part in the green pressed condition. This is accomplished by positioning the

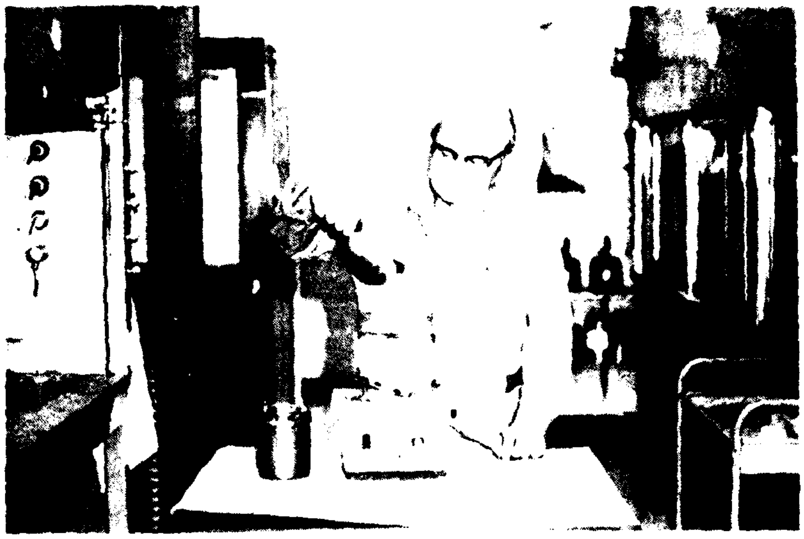


FIGURE 1. LOADING TUNGSTEN POWDER INTO PRESSING BAG

The PVC pressing bag is supported by a perforated metal cage.

FIGURE 2. LOADED BAG ASSEMBLY READY FOR PRESSING

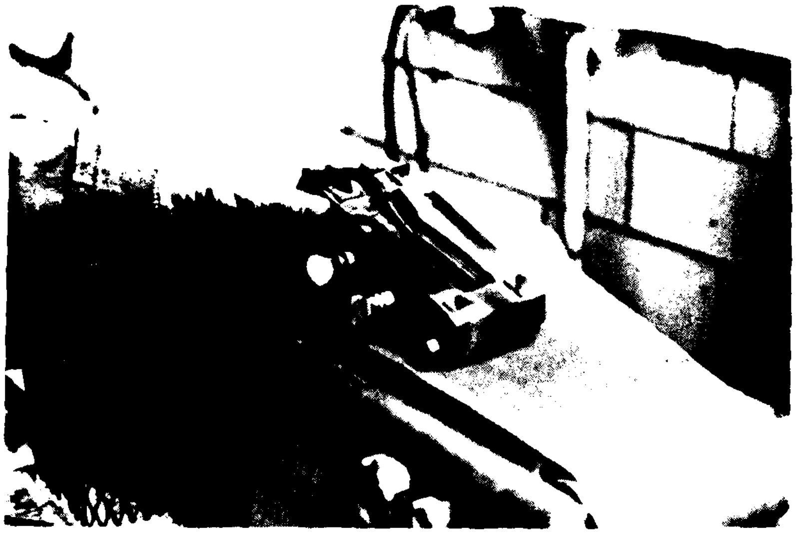
The bag is sealed by two metal bars clamped to the top of the bag by "C" clamps.

part in special fixtures and removal of the excess material with a belt sander. Tungsten carbide inserts in the fixtures act as machining stops to limit the movement of the part into the sanding belt. The fixtures are designed to utilize previously machined surfaces as reference surfaces so that the required dimensional relationships among the different surfaces on the vane are maintained.

The first operation is to remove the high spots from one side of the part (two flat faces). This is done by hand sanding in the fixture as shown in Figure 3. The design of this fixture is shown in Drawing Nos. NWC-003-001, -002, and -003. This is done primarily to minimize any stresses in the green part when it is rigidly held in the fixture used in the next step for machining of the narrow top and bottom surfaces. The design of the fixture for machining the top and bottom surfaces is shown in Drawing No. NWC-004-000. The part being machined is shown in Figure 4. When the desired amount of tungsten has been removed and the tungsten carbide inserts contact the belt, the drag between the part and the belt is reduced and the fixture moves freely on the belt. After the narrow top and bottom surfaces have been machined, the part is placed in another fixture (Drawing Nos. NWC-005-001, -002, -003, and -004) for machining of the flat faces of the vane. The part is supported in the fixture on the same surfaces that had previously been hand sanded. The narrow top and bottom surfaces are rigidly clamped as shown in Figure 5. The two flat faces are then machined by belt sanding as shown in Figure 6. Upon completion of this operation, the components of the fixture are reversed so that the other side of the vane can be machined. The support for the vane is raised by shims (0.050 inch thick) which provide the necessary stock removal on these two flat faces so that the desired final dimensions are obtained. The ends are dressed to remove excess material and the green vane is ready for sintering.

Because of the limited number of vanes produced on this contract at BCL, the fixture for machining the flat faces was designed to handle the machining of both faces. In a high production operation, the machining of each side would use separate fixtures.

FIGURE 3. CLEAN-UP OF TWO ADJOINING FLAT FACES BY HAND SANDING



a. Machining of one of the top surfaces

b. Machining of bottom surface

FIGURE 4. MACHINING OF TOP AND BOTTOM SURFACES USING A BELT SANDER

FIGURE 5. VANE IN THE FIXTURE USED FOR MACHINING OF FLAT FACES

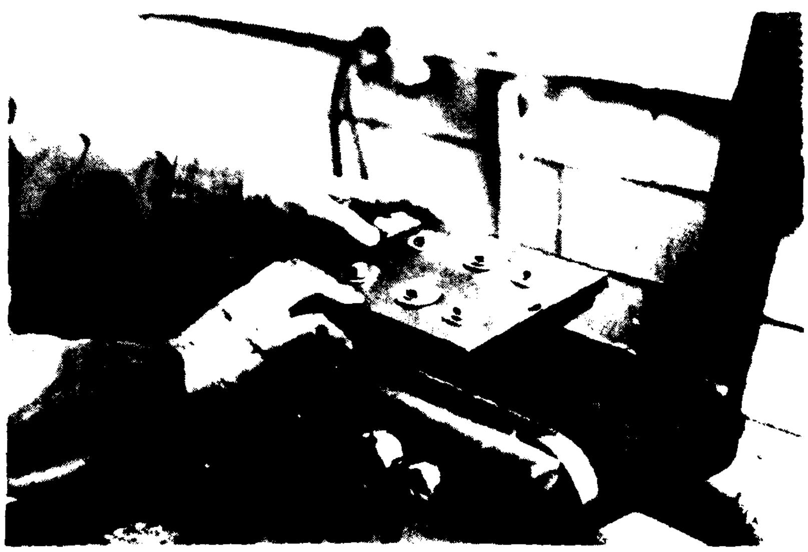


FIGURE 6. MACHINING OF THE FLAT FACES ON THE SIDES OF THE VANE

Sintering

The machined vane is sintered at 1900°C in vacuum for 1 hour. This provides the interparticle bonding within the tungsten structure necessary for strength. The small amount of shrinkage which occurs brings the vane to its final dimensions. This shrinkage during sintering is about 0.020 in./in. After sintering, the weight of the vane is recorded so that the density can be determined. Although a vacuum environment has been used at BCL for sintering, this operation could be carried out in a reducing atmosphere such as hydrogen with comparable results. Minor modifications to the cycle time or temperature may be required to obtain the desired final density.

Infiltration with Copper

The infiltration process is accomplished at 1260-1280°C in hydrogen for a period of 0.5 hour after a hold at 1040°C for 1.5 hours. Prior to infiltration, the exterior of the part with the exception of the bottom surface and an 0.12-inch wide band adjacent to the bottom surface is coated with a brazing stop-off (Wall Colomonoy Corporation, Nicrobraz Green Stop-Off) to prevent rapid coating of the exterior of the part with copper. If, during infiltration, the exterior is coated with molten copper prior to infiltration of the interior, gas trapped in the interior prevents full infiltration. Following the infiltration process, the excess copper at the base of the part is removed mechanically with final clean-up by immersion in dilute nitric acid. Because of the extremely fine pore size of the tungsten, little or no penetration of the acid into the tungsten occurs and virtually no copper is leached from the structure. After the excess surface copper is removed, an immersion density of the infiltrated vane using water is obtained. Since the volume of the vane after infiltration is the same as that after sintering, the sintered density can then be calculated using this same volume. With these two densities, the percent infiltration can then be calculated.

The processing schedule described above has been used successfully at BCL for the infiltration of jet vanes. However, the process as developed at BCL is a batch process, while many industrial processes use a continuous conveyor system through a furnace. Therefore, this process schedule may not be consistent with the type of equipment used in a production operation. Also, the time required for this process in a continuous industrial operation may be appreciably less than that required in this development program.

Final Machining

Although most of the machining of the vane is done in the green condition, certain finishing operations are required. During the sintering operation, the bottom surface does not remain flat. Conventional grinding is used at BCL for machining of the 0.200-inch long flat on the top surface of the vane and to remove stock from the base of the vane to establish the overall height. When the base is machined, the top of the vane and the sides are supported in a fixture that maintains the proper relationship of the two angles in the top surface of the vane to the base surface.

After the base is machined, the 0.501-inch-diameter hole is drilled into it using the same fixture. The hole is drilled with a 0.500-inch-diameter carbide drill and reamed to final size. Following the machining of this hole, the two threaded holes on either side of the large hole are machined using conventional high speed steel tooling.

The ends of the vane are then machined to establish the overall length of the vane. This is done on a belt sander in the same manner as the green machining operations, with the vane held in the fixture shown on Drawing Nos. NWC-006-001, -002, -003, and -004, and in Figure 7. The leading edge of the vane is located with respect to the center line of the large hole in the base of the vane. The trailing edge is then machined to obtain the overall vane length of 4.000 inches. The leading and trailing edges are machined flat using this fixture. The desired radii on these edges are then obtained by a freehand operation using the belt sander. The completed vane is shown in Figure 8.

Inspection

The inspection and test procedures followed during the fabrication of the vane have been covered in a separate document. The Inspection and Test Plan is included as Appendix B.

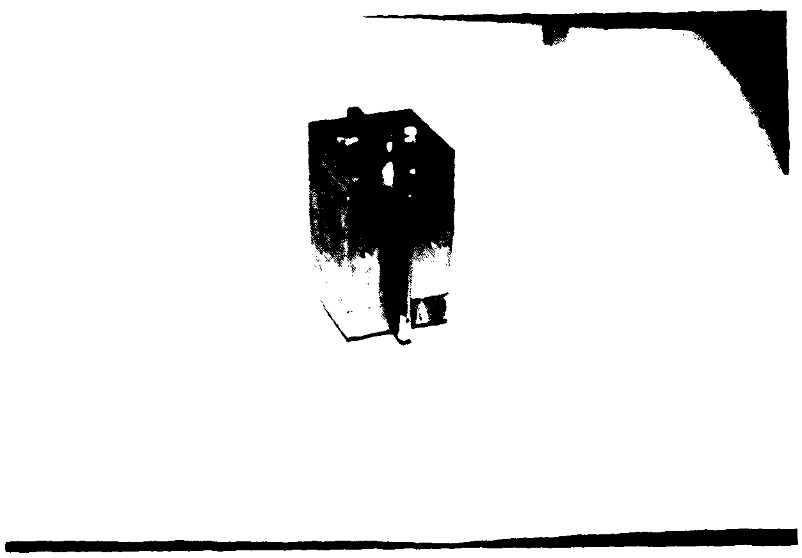


FIGURE 7. FIXTURE USED FOR MACHINING OF THE RADII ON THE LEADING AND TRAILING EDGES OF THE VANE

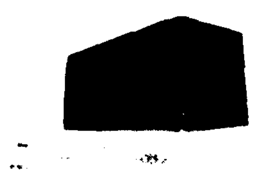


FIGURE 8. VANE AFTER FINAL MACHINING

APPENDIX B

INSPECTION AND TEST PLAN
FABRICATION OF COPPER-INFILTRATED TUNGSTEN JET VANES

An inspection and test plan for the input material and the fabricated tungsten jet vane is given below. This plan has been tailored specifically for the copper-infiltrated tungsten jet vanes produced on this contract and will assure a high-quality final product of uniform properties.

INSPECTION AND TESTING OF STARTING MATERIALS

The input materials for the vane consist of tungsten powder and the copper for infiltration. These materials must meet the following specifications.

1. Tungsten powder
   a. 3.5 - 4.0 micron particle size
   b. Vibratory density of 29 $\pm$ 1 percent of theoretical density
   c. A green pressed density of 76 $\pm$ 1 percent of theoretical after isostatic compaction at 80,000 psi
2. The copper for infiltration will be oxygen-free high-conductivity copper.

These specifications will be verified by the supplier or will be checked prior to use.

INSPECTION AND TEST OF THE JET VANE DURING FABRICATION

During fabrication of the copper-infiltrated tungsten jet vanes, certain quality control, inspection, and test procedures will be followed. These can be divided into two categories: periodic inspections which involve a destructive evaluation of a vane and the non-destructive inspection of the vanes during fabrication. These are each described below.

Periodic Destructive Evaluation

The periodic destructive evaluation of the vanes will be conducted on the first vane of each new lot of vanes produced, at the introduction of a new lot of tungsten powder, and after the fabrication of a given number, e.g., 100, of vanes within a lot of vanes. This inspection shall consist of the following.

1. Metallographic examination of the infiltrated tungsten. The structures will be compared with previous materials that performed satisfactorily during firing.
2. Tensile strength evaluation will be made on two tensile bars machined from an infiltrated vane. The strength must meet the required minimum of 80,000 psi and will be compared to tensile strengths obtained in previous tests to assure that a product of consistent quality is being produced.
3. A complete chemical analysis of the final material will be made to assure that no contamination is being introduced during processing.

Individual Inspection and Test of Each Vane

During the fabrication of each vane, a continuing inspection and verification of procedures will be made to assure that the required quality and properties are being met. The inspection and procedures verification items are listed below. The required information for each item will be recorded on a Data and Inspection Sheet which will accompany each vane during the fabrication process and will be included with each vane at shipment.

1. The pressing pressure used for the green compaction of the tungsten powder will be recorded.
2. The date of green machining of the vane and any unusual observations will be recorded.
3. The vacuum sintering conditions will be recorded.
4. The copper infiltration conditions will be recorded.
5. The sintered density and infiltrated density will be determined and the percent of infiltration determined. The sintered density must be in the range of 75-83 percent of theoretical and the percent of infiltration must exceed 90 percent.
6. Following infiltration, the vanes will be inspected for internal defects by radiography. The vanes will be radiographed at two intensity levels due to the variation in thickness and must meet the following requirements.
   a. Vanes shall not have any voids in excess of 0.040 inch in diameter.
   b. Vanes exhibiting voids in excess of 0.030 inch, whose indicated centers are less than 0.750 inch apart, shall not be accepted.
   c. Vanes indicating linear defects greater than 0.250 inch in length are not acceptable.
   d. Vanes exhibiting transverse voids or other internal defects in the critical region (a 0.75-inch wide zone centered on the hole and extending between the top and bottom surfaces of the vane) of the 0.501-inch-diameter shaft hole shall be rejected.

7. The surface of the vane will be checked for surface finish (not less than 63 AA). Dye penetrant inspection will be used to check for cracks, voids in excess of 0.04 inch, laps, and tears. Any indication of these defects will be cause for rejection. Minor areas of non-linear porosity will be acceptable except in the critical region of the shaft hole. Following final machining, the vanes will be dye penetrant inspected again to check for defects in the area of the shaft hole.
8. The weight of the vane after final machining will be recorded.
9. A dimensional inspection will be made and critical dimensions recorded on an attachment to the Data and Inspection Sheet.

A sample Data and Inspection Sheet with the attachment for the dimensional inspection follows.

COPPER INFILTRATED TUNGSTEN JET VANES
DATA AND INSPECTION SHEET
CONTRACT N00123-80-C-0038

PART NO.

POWDER PARTICLE SIZE

PRESSING PRESSURE

GREEN MACHINING
DATE:
REMARKS:

VACUUM SINTERING
TEMPERATURE
TIME

COPPER INFILTRATION
ATMOSPHERE
SOAK TEMPERATURE
SOAK TIME
INFILTRATION TEMPERATURE
INFILTRATION TIME

SINTERED DENSITY
WEIGHT
VOLUME*
DENSITY

INFILTRATED DENSITY
WEIGHT DRY
WET WEIGHT
WIRE WEIGHT
VOLUME
DENSITY
PERCENT INFILTRATION

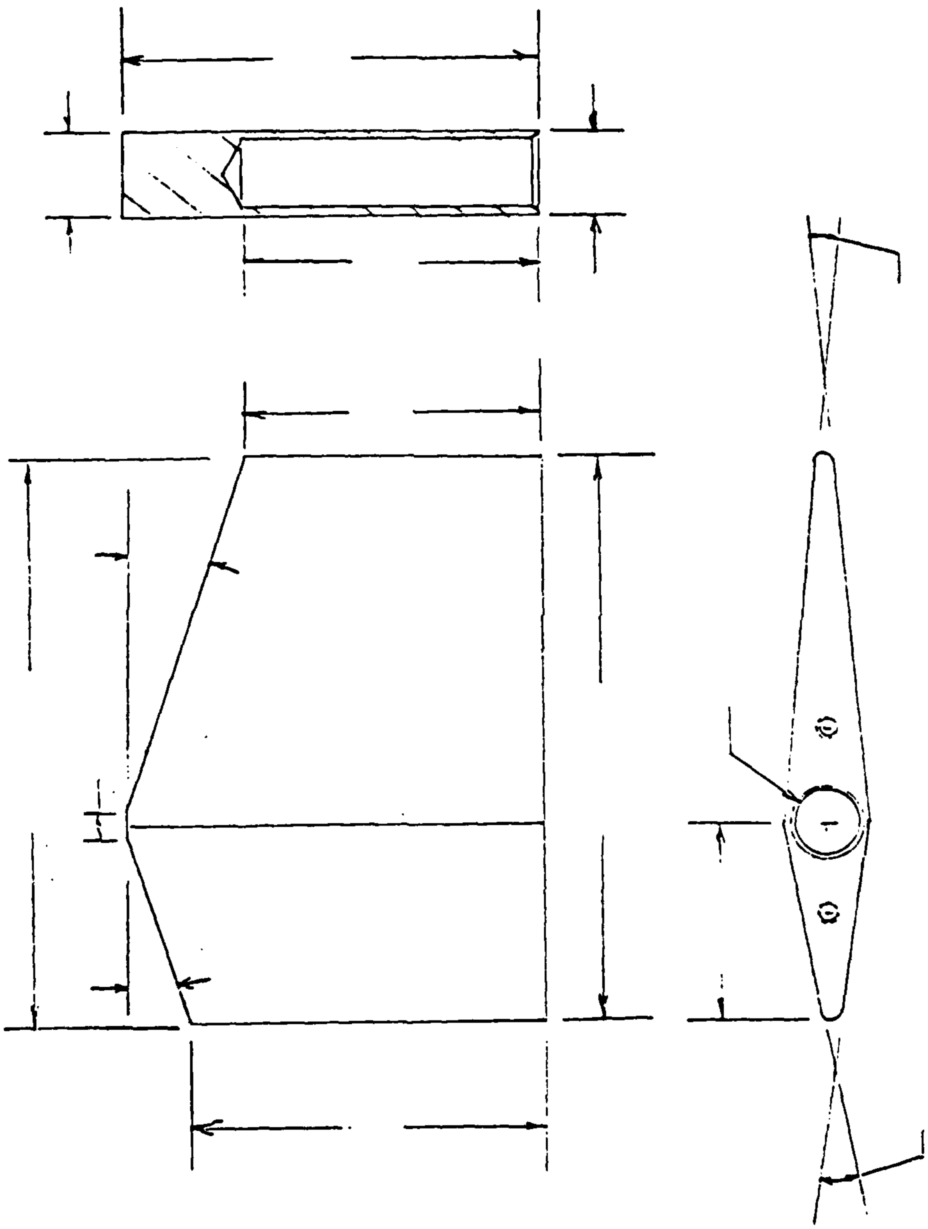
RADIOGRAPHIC INSPECTION
REMARKS:

DYE PENETRANT INSPECTION
REMARKS

PART WEIGHT AFTER FINAL MACHINING

DIMENSION INSPECTION
See Attached Sheet

* Use Infiltrated Volume



Part Number

APPENDIX C

CONFERENCE REPORT

on

MANUFACTURING TECHNOLOGY DEMONSTRATION

FABRICATION OF COPPER INFILTRATED
TUNGSTEN JET VANES

to

NAVAL WEAPONS CENTER
China Lake, California 93555

September, 1981

by

J. H. Peterson and K. E. Meiners

BATTELLE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

Conference Report

on

Manufacturing Technology Demonstration

Fabrication of Copper Infiltrated
Tungsten Jet Vanes

to

Naval Weapons Center
China Lake, California 93555

Introduction

The Naval Weapons Center, China Lake, California, has sponsored a Manufacturing Technology Program at Battelle's Columbus Laboratories to develop a fabrication process for copper infiltrated tungsten jet vanes. These vanes are fabricated from an 80 percent dense tungsten skeleton which is infiltrated with copper. During the development work at NWC to obtain the optimum vane design, commercially available infiltrated tungsten material in the form of a rectangular block was used. This material was machined into the vane configuration with considerable loss of material and machining expense. The objective of the work at Battelle was to develop a process which could produce the vanes at much lower cost.

Program Results

The process which was developed at Battelle reduced the cost of fabrication in two areas. First, by pressing the vane to near net shape, the input material is held to a minimum. Relatively high pressure is used during pressing to impart adequate green strength to the vane to allow machining in the green condition and the density obtained is high enough so that the shrinkage that occurs during sintering is very predictable. Second, by machining the green condition using an abrasive belt, the machining time for most surfaces of the vane can be reduced to 10-15 minutes. Only limited conventional machining is required to complete the vane.

The remaining fabrication steps in the vane fabrication process, such as sintering and infiltration, are handled using conventional practice for this material. Inspection of the vanes indicated that high quality vanes which met the contract requirements were produced by this process. Actual firing tests of these vanes indicated their performance was equal to or better than those previously evaluated.

Technology Demonstration

In order to acquaint interested personnel from the Navy and industry with this process, a technology demonstration was held at BCL on September 15, 1981. Invitations to this demonstration were sent out by NWC to personnel within the Navy and to industrial firms suggested by BCL. An agenda for the demonstration was prepared at a joint meeting of NWC and BCL personnel on August 20, 1981. This agenda is given below:

AGENDA

Technology Demonstration

Contract N00123-80-C-0038

Fabrication of Copper Infiltrated Tungsten Jet Vanes

September 15, 1981

8:30 - 9:00	Coffee	
9:00 - 9:15	Introduction to Battelle	K. Meiners
9:15 - 9:45	Program Introduction & Summary	M. Ripley-Lotee
9:45 - 10:45	Process Description	J. Peterson
10:45 - 11:00	Break	
11:00 - Noon	Display of Process Equipment and Product	J. Peterson
Noon - 12:30	Test Results	
12:30 - 1:30	Lunch	
1:30 - 2:00	Discussion	
2:00 - 2:15	Closing Remarks	M. Ripley-Lotee
2:15 - 3:30	Tour of Battelle Facilities (Optional)	

The Technology Demonstration was attended by eight industry representatives and two Navy personnel. In addition, three BCL and two NWC representatives, who were involved with the program, were present. A list of the attendees is attached to this report.

The presentation was well received and genuine interest from the industry representatives was expressed concerning the manufacture of the vanes for the Navy. Discussions with these representatives after the presentation indicated that the type of process developed could be readily adapted into their normal production. Since the presentation, two of the companies have contacted Battelle to obtain additional information.

As a result of the technology demonstration, industrial firms with the capability of producing copper infiltrated tungsten jet vanes for the Navy have been made aware of the need for these vanes and exposed to the process for fabricating the vanes. A number of these firms showed definite interest in producing these vanes. The fabrication process developed in this program is readily adaptable to the normal production operations of these companies and the transfer of this technology should be made without difficulty.

ATTENDANCE

Industry Briefing
Contract N00123-80-C-0038
Fabrication of Copper Infiltrated Tungsten Jet Vanes

September 15, 1981

Battelle-Columbus Laboratories

Name	Company
Hiram Cox	Rockwell International
Robert Fitzgerald	Chandler Evans
Tom Hogen	Hogen Industries
John R. Thompson, Jr.	Naval Surface Weapons Center Dahlgren, VA
William J. Welsh	Naval Materials Command Ind. Resources Detachment
Richard Hulbert	Teledyne Firth Sterling
David Fleckenstein	CMW, Inc.
Henry P. Utzinger	Kulite Tungsten Corp.
Rossa W. Cole, Jr.	Teledyne Powder Alloys
James J. Kuznick	Powder Tech. Co.
Kenneth E. Meiners	Battelle Columbus Laboratories
James L. McCall	Battelle Columbus Laboratories
James H. Peterson	Battelle Columbus Laboratories
Michael Ripley-Lotee	Naval Weapons Center
Daniel G. Pacquin	Naval Weapons Center

Naval Weapons Center
Code 3273
China Lake, CA 93555
Attn: M. Ripley-Lotee or S. O'Neil
(714) 939-7224/7378

APPENDIX D

TEST REPORT: MOTOR STATIC FIRING TESTS

The ultimate test of any jet vane, and the final determination of a particular design's acceptability, is a motor firing test; i.e., does the vane survive the motor exhaust environment with acceptable performance for the required period of time. Consequently, three static firings were conducted by NWC using manufacturing technology (Battelle) vanes from the experimental, prototype and preproduction lots. Each test was for: (1) lot acceptance, (2) evaluating the performance of the Battelle jet vanes, and (3) comparing Battelle vanes with vanes manufactured using the standard process.[1] Although a different motor was used in Test 2, the test configuration was similar to that depicted in Figure 1 (Test 3) with the vanes attached to heat shields and fixed in the trailing position.

TEST NO. 1

The first static firing was conducted with one of the three Lot 1 vanes to obtain an indication of vane performance before proceeding with Lot 2 production. The vane was fixed in the motor exhaust along with a standard vane for comparison.

The motor was a low-cost controllable booster (LCCB) that could use jet vane control. Nominal performance for the nonaluminized HTPB propellant-loaded motor was 10,500 pounds of thrust and 5.4 seconds burn time.

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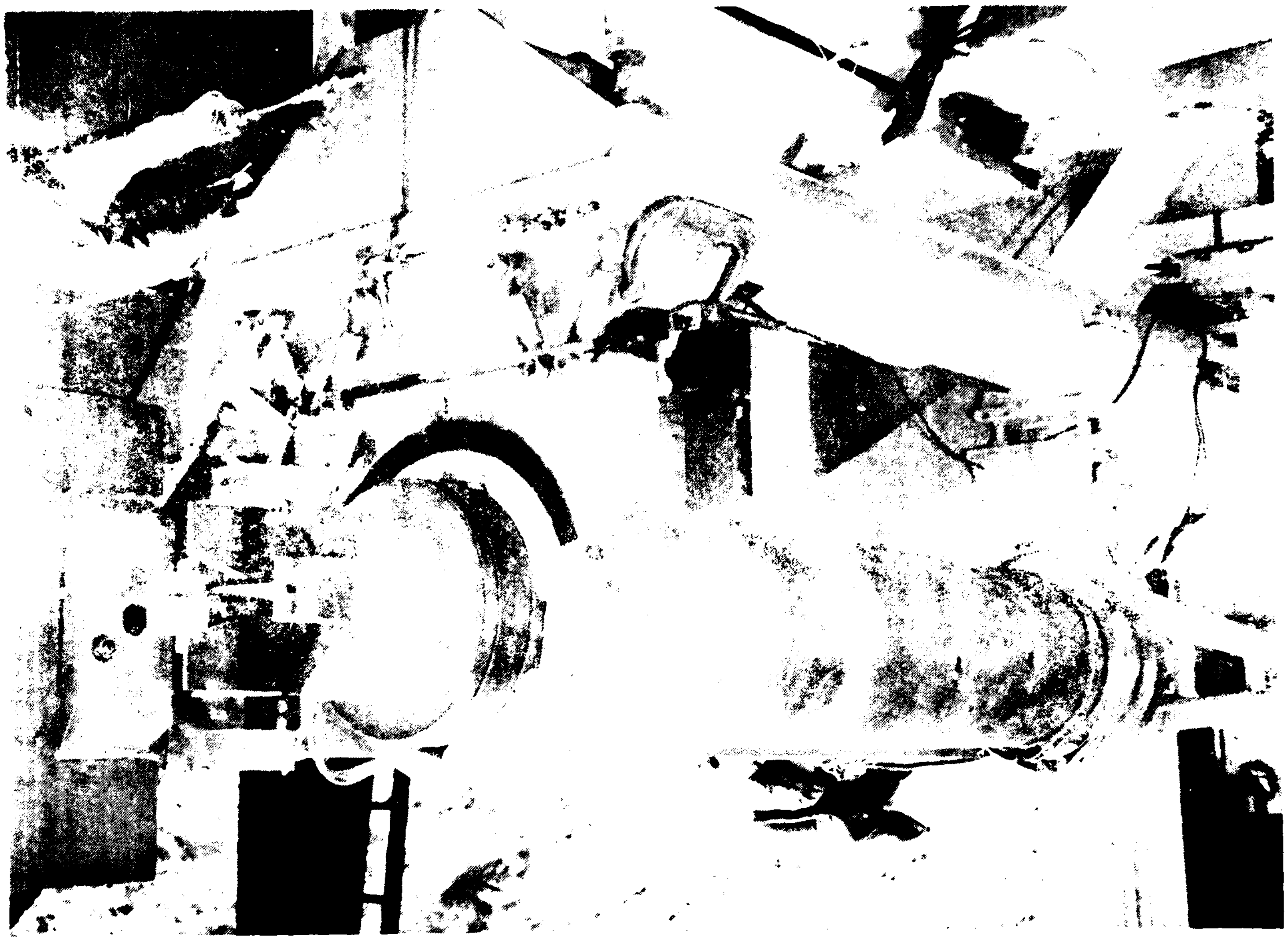
1 Vanes referred to as standard were manufactured by Teledyne Wah Chang from a simple mill block of sintered and copper-infiltrated tungsten.



Performance and post-test appearance of the vane were nearly identical with the control vane. Following the test, the vanes were sectioned and subjected to metallographic examination. Figure 2 shows the vane sections selected for examination. Vane B3 is the manufacturing technology vane; vane WC the control vane. Figures 3 through 6 show the microstructure of selected regions for both vanes. The figures reveal a more complete and uniform copper infiltration of the tungsten skeleton for the manufacturing technology vane. A scanning electron microscope (SEM) micrograph and elemental dot map of the copper infiltrant material are shown in Figure 7. Figure 8 shows the pattern and effects of localized heating on the grain structure of the shaft.

TEST NO. 2

The second test was conducted to compare Lot 1 and Lot 2 manufacturing technology vanes with control vanes in a long duration all- boost motor environment. This test not only compared vane performance, but also evaluated techniques directed toward increasing vane endurance.

The motor was a modified SMARTROC case cast in an all-boost configuration with nonaluminized HTPB propellant. Total impulse was approximately twice that of the motor in Test No. 1. Burn time was 8.5 seconds and average thrust was 14,000 pounds.

Two standard vanes were used as controls for comparison against one each of Lot 1 and Lot 2 manufacturing technology vanes. In addition, the forward two-thirds of the Lot 2 vane was coated with zirconia to reduce heat transfer to the shaft. With the exception of one control vane, all vanes were fixed to the shaft with a single pin through the back of the vane. The heat shields were attached by two screws. The screw forward of the shaft was of tungsten; the aft screw, high strength steel. The unique mounting scheme for the one

[illegible] 1, VANES.

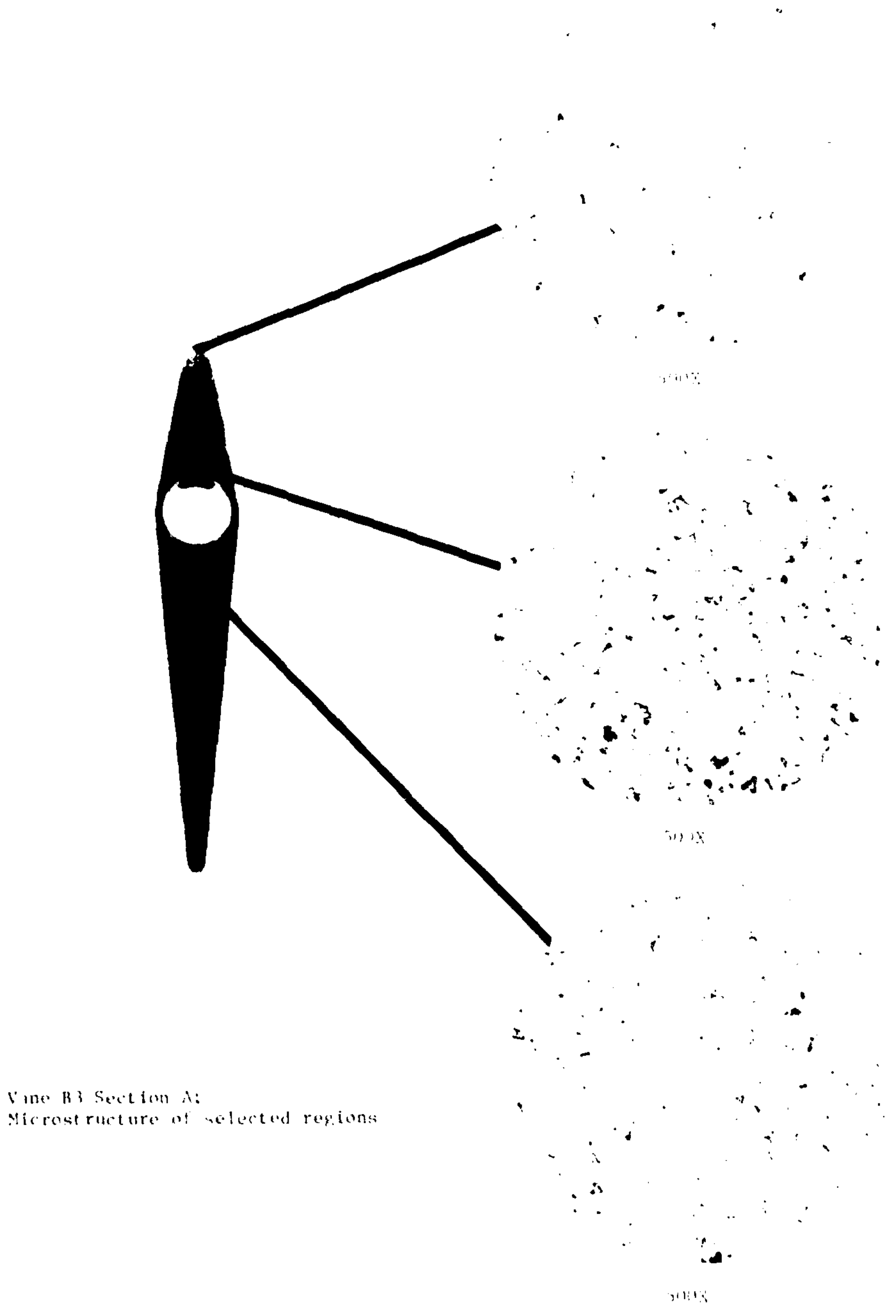


FIGURE 3. TEST NO. 1, CONTROL VANE MICROSTRUCTURE OF SECTION A.

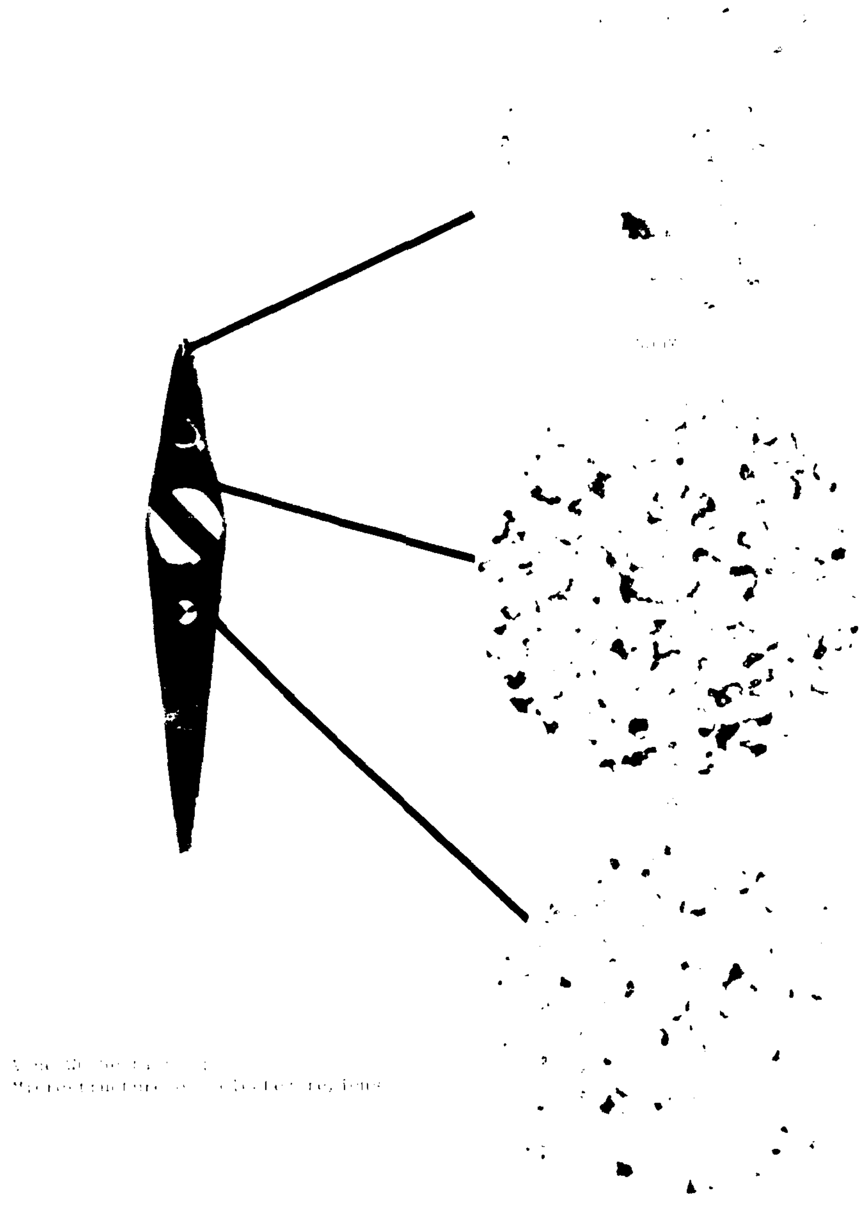


FIGURE 4. TEST NO. 1, CONTROL VANE MICROSTRUCTURE OF SECTION B.

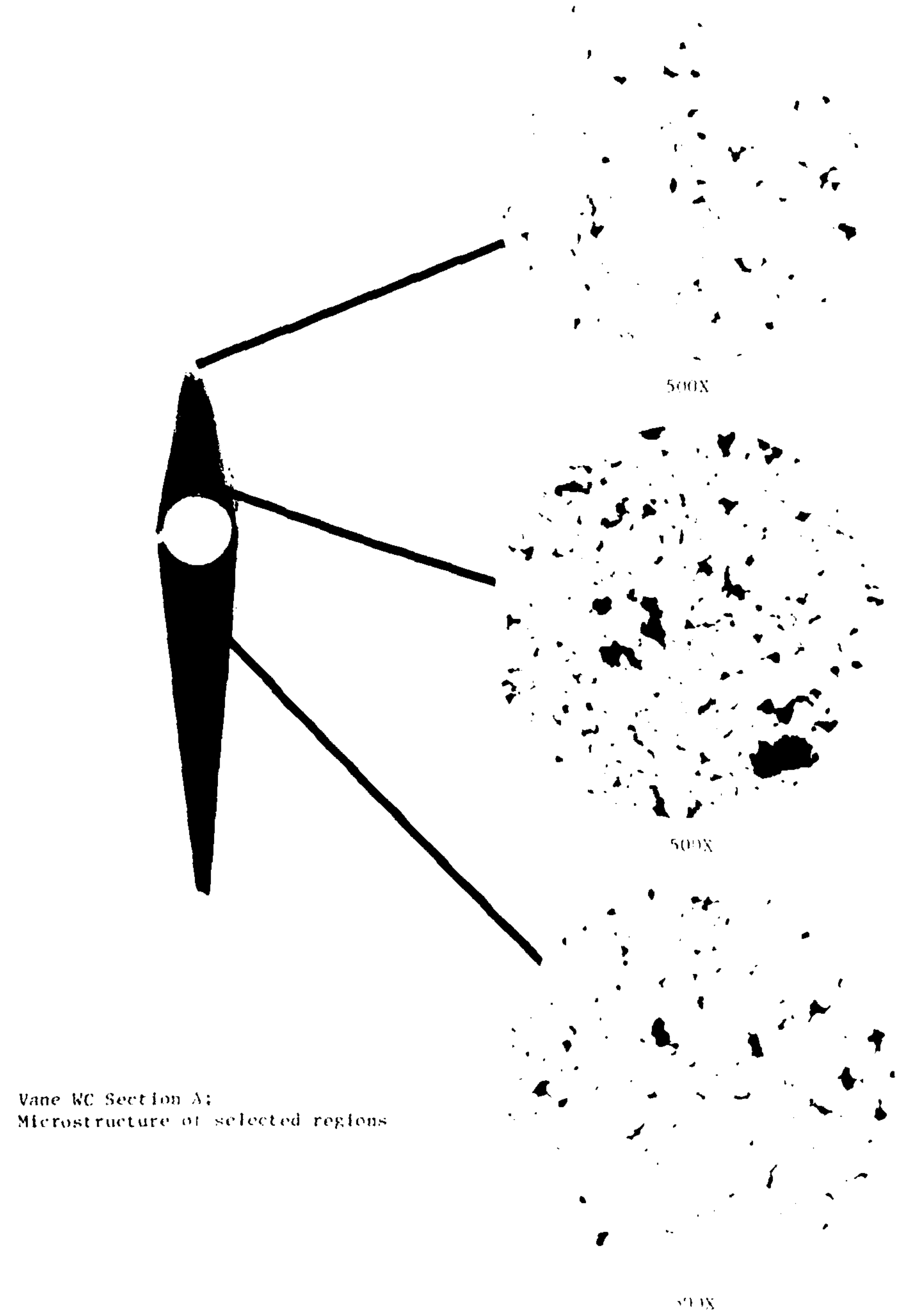


FIGURE 5. TEST NO. 1. MANUFACTURING TECHNOLOGY VANE MICROSTRUCTURE OF SECTION A.

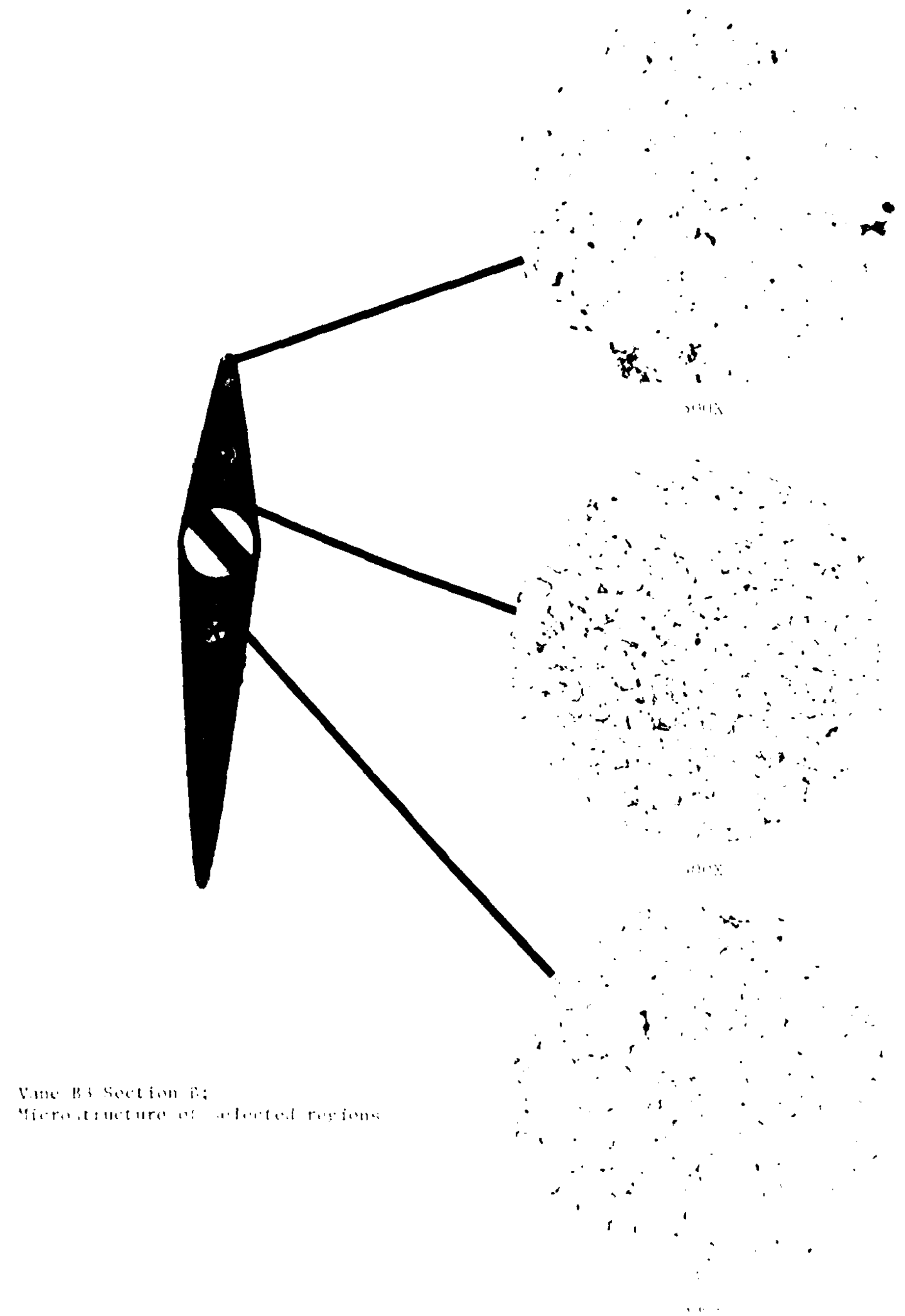


FIGURE 6. TEST NO. 1, MANUFACTURING TECHNOLOGY VANE MICROSTRUCTURE OF SECTION B.

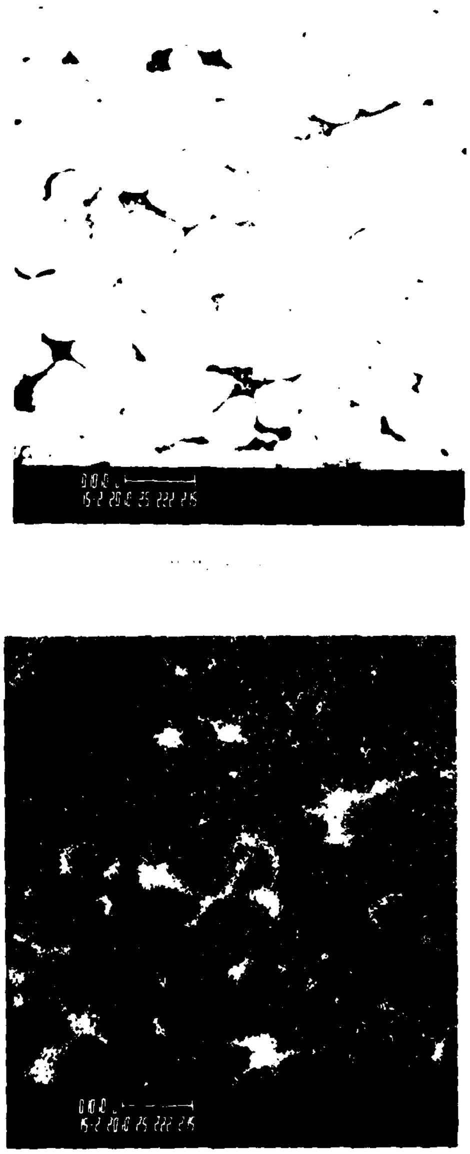


FIGURE 7 SEM MICROGRAPH AND ELEMENTAL DOT MAP OF COPPER INFILTRATED TUNGSTEN

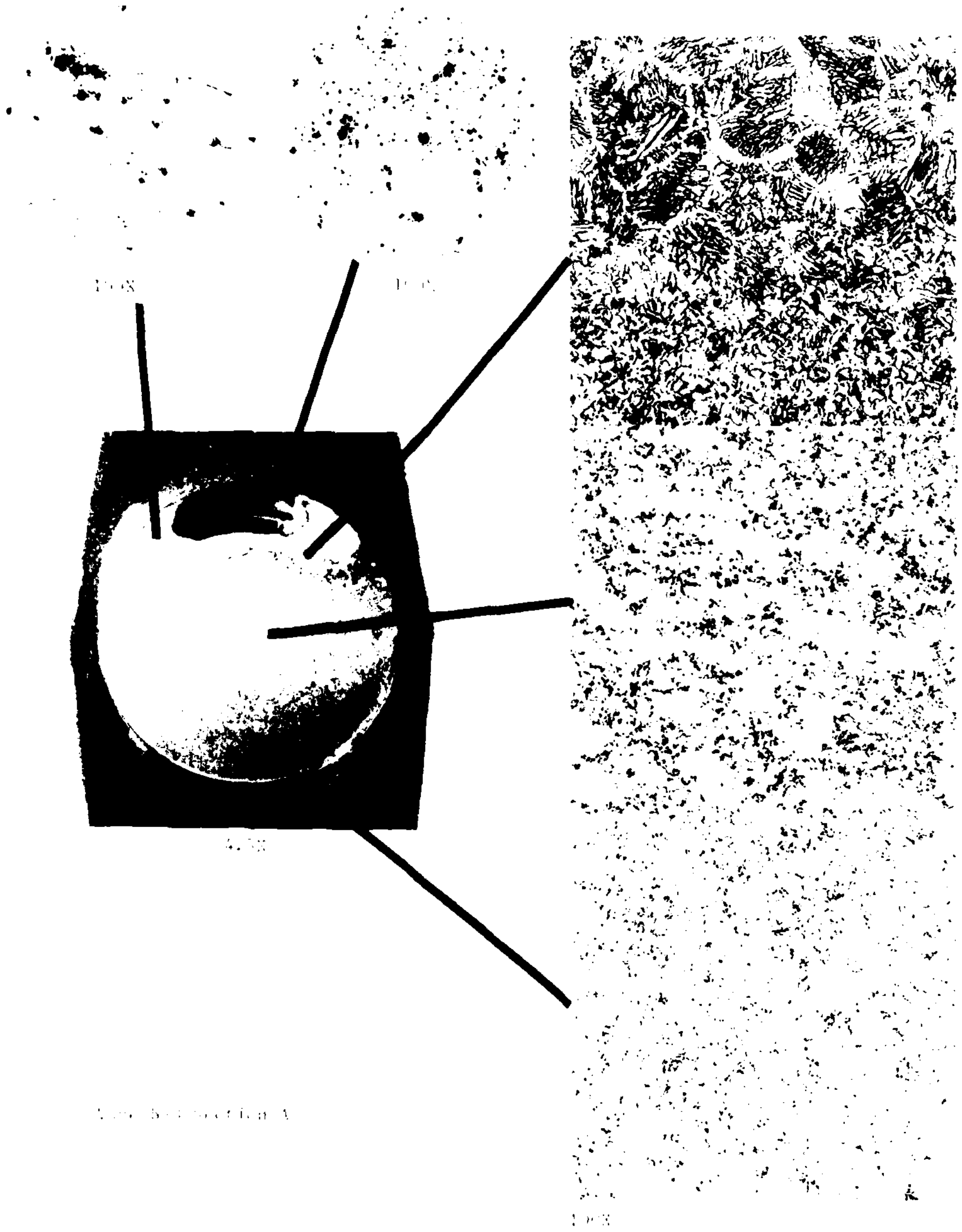


FIGURE 8. HEATING EFFECTS ON GRAIN STRUCTURE AND COPPER INFILTRANT FROM TEST NO. 1

control vane consisted only of the two screws which passed through a collet on the shaft, thus eliminating the need for the pin and a potential heat transfer path into the shaft.

Both control vanes failed 7.5 seconds into the test. The vane without pins failed due to fracture of the forward screw, which allowed the vane to set back from the heat shield, stressing the aft screw to failure and releasing the vane. The other control vane separated due to shaft failure. In this case, failure was due to vane fracture around the shaft, which allowed direct impingement of the exhaust gases on the shaft.

Both manufacturing technology vanes survived the test. Figure 9 shows the failed control vanes in the foreground and the Battelle vanes still attached to the heat shield in the background, with the zirconia-coated vane on the left. The zirconia-coated vane showed very little erosion, and that by melting and subsequent flow off, rather than by spalling off. The condition of the shaft was excellent, possibly the best of any in the test series to date. Although the other Battelle vane survived the test, the forward screw incurred a fracture, which resulted in shaft erosion due to setback. The screw fracture was probably caused by the large thermal gradient through the screw and the increased notch sensitivity resulting from the sharp threads. Since this problem can possibly be corrected by a design change, it appears that an uncoated vane could satisfactorily survive the full duration of an all-boost motor, of the type fired, with an acceptable margin of survivability.

Following the test, the vanes were sectioned and analyzed. Again the manufacturing technology vanes exhibited a finer grain structure and a complete, more uniform infiltration of the tungsten.

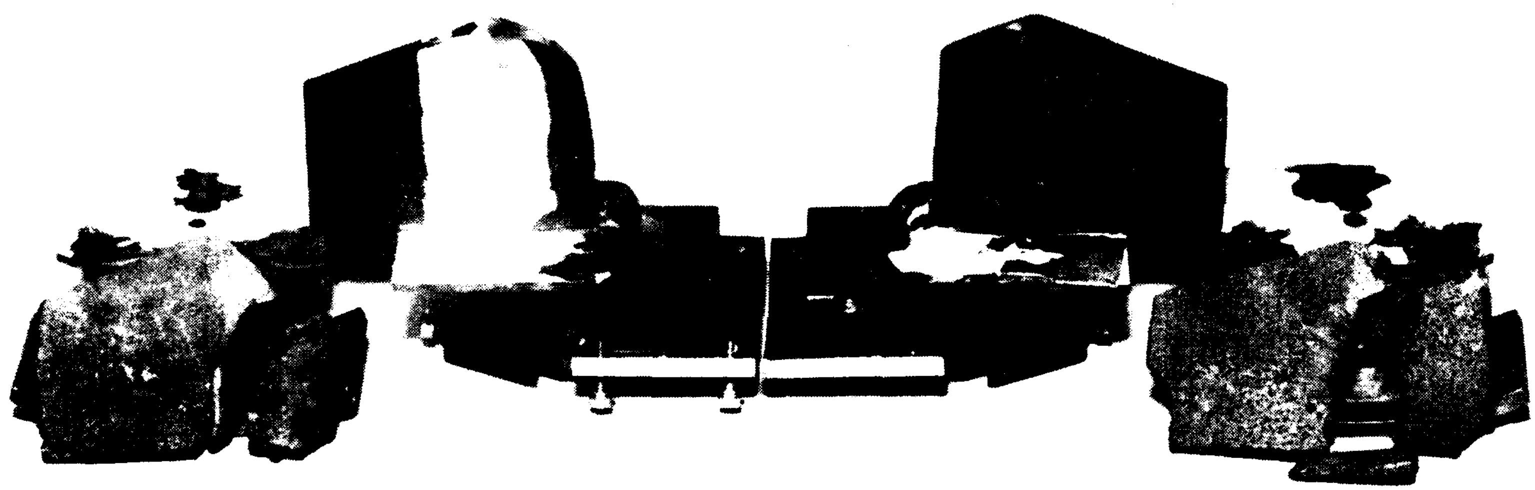


FIGURE 9. VANE TE[illegible] VANE WITH STANDARD VANE IN [illegible]

TEST NO. 3

The third test was conducted as a lot acceptance test of Lot 3 vanes. Two vanes from Lot 3, one from Lot 2, and one control vane were used. The test set up was otherwise identical to Test No. 1.

All vanes performed nominally. The manufacturing technology vanes showed minimal erosion; the control vane erosion was normal. The test was considered highly successful and verified acceptability of the Lot 3 vanes.

SUMMARY

The static firings demonstrated the performance of the Battelle vanes and showed them to be at least as good as the baseline vanes. The tests also verified that the improved manufacturing process developed by Battelle is an acceptable means of producing copper-infiltrated tungsten jet vanes at comparable or higher quality and lower cost than previous methods.

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